

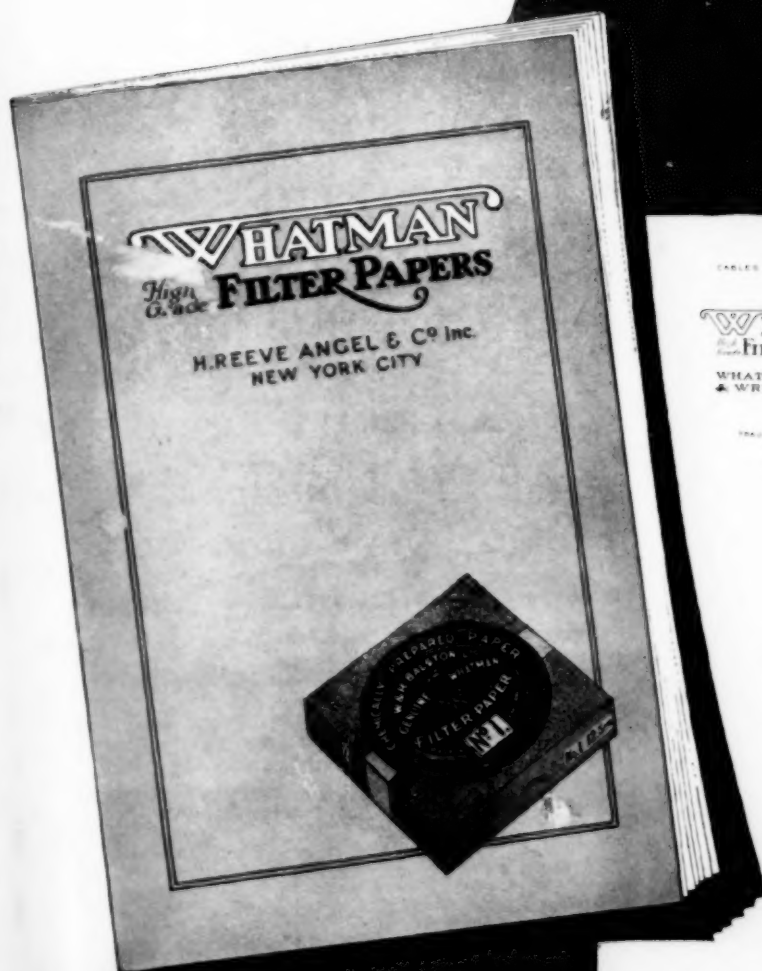
*A Semimonthly Technical Newspaper*

# Metallurgical & Chemical Engineering

New York, February 15, 1917

McGraw Publishing Co. Inc.

Vol. XVI, No. 4 25c a copy



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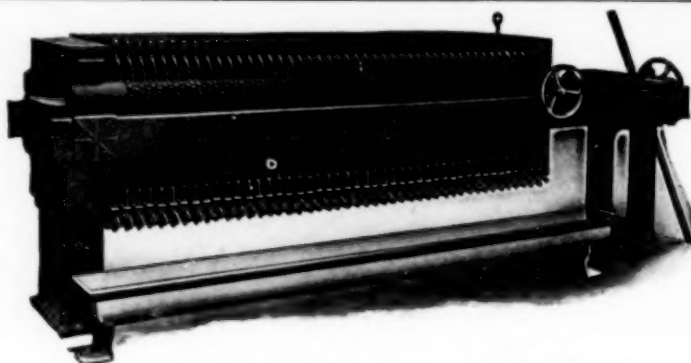
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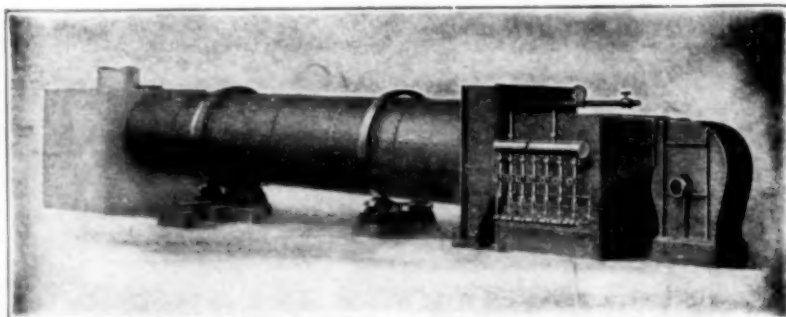
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# Metallurgical and Chemical Engineering

A Consolidation of  
ELECTROCHEMICAL AND METALLURGICAL INDUSTRY and IRON AND STEEL MAGAZINE

Vol. XVI

NEW YORK, FEBRUARY 15, 1917

No. 4

PUBLISHED SEMI-MONTHLY ON THE FIRST AND FIFTEENTH  
OF EACH MONTH BY THE

**McGraw Publishing Company Inc.**

JAMES H. McGraw, President.

A. R. CLIFFORD, Secretary.

JOHN T. DeMott, Treasurer

239 West 39th St., New York.

TELEPHONE. 4700 BRYANT. CABLE ADDRESS. METCHEM, NEW YORK  
ROCKY MOUNTAIN OFFICE.....401 Boston Bldg., Denver, Col.  
PACIFIC COAST OFFICE.....Rialto Bldg., San Francisco, Cal.  
CHICAGO OFFICE.....Old Colony Building  
CLEVELAND OFFICE.....Leader-News Building  
PHILADELPHIA OFFICE.....Real Estate Trust Building  
LONDON OFFICE.....Hastings House, Norfolk St., Strand

E. F. ROEHR, Ph.D., Editor.

S. FISCHER, Ph.D., Western Editor.

J. MALCOLM MUIR, Manager.

Yearly subscription price for United States, Mexico and  
United States dependencies, \$3.00; all other countries, \$4.00.  
Single Copy 25 Cents

When change of address is ordered, the new and the old  
address must be given. Notice must be received at least 10 days  
before the change takes place. No back copies for more than three  
months.

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Entered as Second-Class Matter at the Post Office at New York,  
N. Y., under the Act of Congress, March 3, 1879.

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## The Niagara Falls Power Famine

A un point de vue plus objectif il est bien évident que cette  
crise de la houille blanche à Niagara Falls est l'indice que  
les Etats-Unis sont beaucoup plus pauvres qu'on ne se  
l'imagine en forces hydrauliques. Et il ne faudra pas perdre  
de vue plus tard cette indication quand on étudiera l'orienta-  
tion de certaines fabrications électrochimiques.

Thus the *Journal du Four Electrique* in its issue of  
Jan. 1, 1917, and we would respectfully submit the re-  
mark to the distinguished consideration of the eminent  
gentlemen in Washington who will be so largely respon-  
sible for the future of electrochemistry in the United  
States.

At this writing the Cline bill has passed its third read-  
ing and the permanent legislation in regard to Niagara  
power which it proposes includes, of course, the inevita-  
ble tax on the water used. Equally inevitable appears  
the fate of the bill when it goes to the Senate, which will  
refuse consent to the tax. This means amendment fol-  
lowed by conference and then nothing will be done.

Meanwhile, with the nation facing the probability of  
war, munition plants and all kinds of industries, abso-  
lutely necessary to a country at war, dependent on the  
products of the Niagara Falls electrochemical industries,  
must suffer because the eminent gentlemen in Washing-  
ton know the value of the monopoly scare as a vote  
catcher.

The temporary relief measure passed by Congress is  
not of the very smallest benefit to the United States. In  
the first place, ice is not a satisfactory source of power,  
and at the present time weather conditions are such that  
the additional water is in this unavailable form. Sec-  
ondly, when the weather improves conditions will be far  
worse than before owing to Canadian demands. Canada  
knows what war means and hence the demand for 50,000  
hp. by April 1st will be insisted on and will be obtained  
by the simple process of cutting off this amount of power  
from the United States. Finally, unless some unfore-  
seen event changes present Canadian developments no  
power whatever will be exported from Canada to the  
United States by the end of this year.

Let us consider what this means. According to fer-  
mer legislation 160,000 hp. could be exported from Can-  
ada to the United States. Our electrochemical indus-  
tries counting on this import and the normal improve-  
ments in the efficiency of hydro-electric developments  
have built their great plants and made themselves a vital  
necessity to the whole country. Now in the time of our  
need we have a power famine which in a few months  
will be infinitely more serious than at present. Of the  
160,000 hp. formerly available not a mouse-power will  
come from Canada.

The additional 4400 cu. ft. ought to give us 80,000 hp.



if developed at high efficiency and the water now used would give an enormously increased power were it used in the efficient manner made possible by the modern improvements in hydro-electrical engineering. None of this, however, is possible so long as the inept bungling of legislators prevents the necessary developments. As far back as 1906 when the infamous Burton Act was passed the Niagara Falls Power Company had all its plans completed for finishing its great pioneer work begun in the last decade of the nineteenth century. Had it not been for that Act, Niagara Falls would long ago have had from this development 200,000 hp. in a plant showing the highest attainable efficiency. Even had some common sense penetrated the legislative mind in 1909 when the Burton Act expired, this work would have been undertaken, but the monopoly-scare mongers were busy and all thoughts of efficiency and the conservation of our resources from a strict engineering standpoint as well as from the broader viewpoint of national industry were disregarded.

It cannot be too often or too strongly insisted that we shall never get the full efficiency out of the water taken from the Niagara River until permanent, common-sense legislation warrants the great expense involved in taking advantage of modern hydro-electric engineering principles and methods.

Once more with war threatening us and the country looking to Congress for legislation that will put us in the strongest position to face its strain as well as that of the economic struggle which will follow it, our legislators have an opportunity to retrieve the disastrous blunders of the past, blunders which have caused a power famine daily increasing in intensity and which the whole country will feel before many months are past. Will this opportunity be let slip for the sake of collecting a small revenue or to please the dupes of the monopoly-scare monger? Every cent of a tax of this kind is simply a hindrance to the attainment of the ideal in the efficient use of water power and is surely borne by the electrochemical industries and those dependent on their products. For the purpose of collecting a small revenue the larger indirect revenue to be derived from the industries supplied by the hydro-electric power is sacrificed and the welfare of the whole country injured to an extent that it is impossible to estimate.

Truly the Washington efficiency engineer has a single eye to political conservation.

### Tonnage of Iron in Future

It has always been difficult to believe that the rate of growth previously maintained in the American iron and steel industry could be maintained in future. The fact that the growth in tonnage had been in geometric ratio was in itself suggestive that it could not continue in the same fashion. In the years immediately following the panic of October, 1907, it was felt in many quarters that at last the time had arrived for the rule to break down, that indeed it had broken down. The large tonnages lately produced, however, have practically put the rule in operation again. As shown in

these columns Sept. 15, 1916, pig iron production in the decade ended Dec. 31, 1915, compared with output in preceding decades, was very nearly in conformity with the old rule of a doubling every ten years. In 1916 the output was much larger, and 1917 promises a gain, where a comparison made by using the decade ending with the present year will show a still closer conformity.

We can look into the future with clearer vision than one decade or two decades ago, because we have more history to guide us. The increase in the use of steel and iron is no longer mysterious. When mild steel was a new thing a structure made of it was regarded as a permanent work, not altogether like the pyramids, perhaps, or like the iron pillar at Delhi, now nearly if not quite 3000 years old and still "going strong," but at any rate something about which it was reasonable to wonder how long it was capable of lasting. Now we know it is more likely to be a question of how long men will continue to need it. Skeleton steel buildings only ten years old have been torn down to make way for much taller structures with much heavier members.

For the consumption of iron per capita to remain the same it would, of course, be necessary for the population to increase at the same rate as the production. This, of course, it has not done. If the population growth were in geometric ratio it would not be in as great a ratio, and the per capita consumption would have to increase. Population growth, however, has not even been in geometric ratio. It was so, approximately, from 1790 to 1860, the increase per decade being about 35 per cent, but in the three following decades the increase was only about 26 per cent, while the decennial increase from 1890 to 1910 was still smaller, 21 per cent.

The production of pig iron per capita was about 94 lb. in 1870, 330 lb. in 1890, and 670 lb. in 1910, while it is now about 930 lb., taking only the population of continental United States.

It is really a question of the needs and purchasing ability of the people. The wealth of the United States doubles in a little more than a decade. The freight ton-mileage of the country has been doubling about once in every dozen years.

The rapid growth of the automobile industry is often cited as an illustration of the expansion in the use of steel. We beg to use the illustration of the automobile industry just the other way. When people buy 1,500,000 automobiles in a year at, say, \$800 apiece, they are spending nearly a dollar for every pound of iron thereby absorbed, an enormously expensive way to provide an outlet for iron. It is no illustration at all. Rather one should view the case that the people are rich enough so that more than a million new owners a year appear, and soon they will be well provided and part of the money will be spent otherwise.

The laying of petroleum pipe lines, which has consumed more than a million tons of iron and steel, did not noticeably interfere with the growth of rail freight traffic, nor will the automobile. The past few years



have been bad with the railroads, from too much saying by state and federal governments of what they must not do, but soon, there is good reason to hope, will come the time when the government will tell the railroads what they must do by way of expansion to meet and anticipate the needs of the country, and will assist them, if necessary, in the expansion. The advent of the steel freight car and heavier locomotives forced the railroads, ten years and more ago, to relay nearly all their track with heavier rails. Now still heavier cars and locomotives are coming, and when the scruples of the railroads as to the quality of a 125 or 150-lb. rail, as compared with an 85-lb., are removed, another era of rail replacement will come. Then there is electrification, no longer a technical problem, but almost wholly a financial one and commercially this will be perhaps the biggest development of all; and we may add in parenthesis, it may affect the copper industry even more than the steel industry.

There are few uses for steel in which a large expansion in tonnage cannot with confidence be predicted. Most of these lines of consumption, hydroelectric development as a conspicuous instance, are in their infancy as compared with railroading, which promises heavy consumption in future.

It has been suggested that as time passes and the amount of iron and steel in use increases, the amount to be won from the ground by ore mining will not need to increase so rapidly, as more scrap will be returned after use. It is a question of mathematics. If the proportion of the production that will eventually come back, as against that which is totally lost by oxidation and comminution or dispersion or whatever it may be called, remains constant, and the average period of life remains constant, then with a geometric rate of increase in consumption the rate of increase in production will not change. If production doubles each ten years, and one-half comes back as scrap in ten years, then the scrap will always be one-fourth the production. If it is twenty years instead of ten the proportion will always be one-eighth. If the period of employment lengthens, the proportion of scrap to supplement new production will decrease. If the period of employment decreases, the smelting of iron ore will increase by smaller ratios, but the activity in reworking will increase.

### On the Blindness of Others—and Ourselves

The other day a woman who ran an army canteen in England last year related how, with the problem often before them to feed several hundred soldiers on absurdly short notice, no labor saving devices were at hand, no dish-washers or bread-cutting or sandwich machines were available; in fact, there was nothing provided to save work.

In John Masefield's book on the ships of Nelson's day it is noted that 'eart of hoak and other lumber was seasoned in water and that the British ships were heavy and damp and unwholesome in consequence; yet they would not change their methods. The Yankee clipper ships were built on French models and were far more

speedy and desirable, but nothing could drive into the heads of those British shipbuilders the idea that it is better to season wood than to soak it.

Before these days of blood and anguish a very distinguished American engineer went through the Krupp works at Essen. With great pride they showed him seventeen men at a job successfully accomplishing what it takes three men to do in Pennsylvania, and in another place a military company of marching men were doing the work of a chute.

If we only close our eyes to the inward glance; if we address ourselves to looking very hard at other people and neglect the thought of our own short-comings, it does seem as if those foreigners were dunderheads for fair. This is a habit with us. We may admit that we have single faults but other people are promiscuously and miscellaneously wrong. Ask any Democrat what he thinks of the Republicans and he will say they have no character and no ideals, and the Republican will say of the Democrats that they have no sense. We are right and the other fellows are wrong.

Now we respectfully submit the gospel of modesty. We can give plenty of reasons but we can give no excuse for our beehive coke ovens. We can explain the wretched condition of our army, but that does not excuse it. We can explain how some industries use up men and take the hearts out of them whereby discontent and disorder and hatred and malice are caused to thrive like a green bay tree, but that does not excuse them. We can point with pride to the expedition with which living shacks for workmen have been constructed, but if they are unwholesome or even ugly we should do better to recall the pride, send for a sanitary engineer and get busy.

In the big vision of things explanations do not excuse. The sooner we get this into our heads and hearts the better it will be for us. All the world's a stage—it's a regular exhibition, and all our works are on show. We cannot attach our personal labels to them, however, whether we want to or not, for more than a very little while. Our works are on show as the product of the people of the United States and that is the only label that will stick. The people of the United States have fallen short in many ways, and all of us have had a hand in it.

The whole world is in a very bad way and most of the children of the earth see red and red only. Now Crile and many others have shown the inwardly destructive force of anger, and by this a great part of the world is being crippled in mind as well as in body. We shall have that to meet, and to avoid, if we would keep our heads, no matter how much fighting we may have to do. What we need above all things to-day is the philosophic vision that looks for the right, that sees our own faults and tries to remedy them. It is always bad diplomacy to point out their faults to other peoples. They have to find them out for themselves by reason or by force of circumstance. Talking will do no good. But our own lapses are excellent subjects for us to consider. They are not such pleasant subjects as the faults of others, but they are vastly more wholesome.

## Readers' Views and Comments

### The Flotation Suit Decision

*To the Editor of Metallurgical & Chemical Engineering*

Sir:—A very interesting letter signed "A. B. C.," appears in Feb. 1 issue of your journal re the legal status of flotation. One can agree with the digest of the writer and yet not accept his conclusions that the Supreme Court has made any mistake or is likely to change its mind.

All agree that the U. S. Patent Office is not what it was intended to be, and that litigation is of such a nature and expense that the inventor has little chance, and only when large capital undertakes the defense of a patent, as in the flotation cases, can the patent get a fair hearing.

The flotation case is one of the first instances where industry has been checked by the courts, and forced to recognize an invention that it adopted and used in defiance at immense profit.

The argument of "A. B. C." as to the academic knowledge previous to U. S. Patent No. 835,120 was all known and admitted, the commercial use of any of this knowledge was non-existing, yet there was great commercial necessity to find a commercial process. The court practically disposed of all this previous art under the fair assumption, knowing the conditions, that none of it was commercial.

If it was an obvious engineering feat to find a minimum and commercial percentage of oil, it had not been done, and the courts decide it was thus not obvious.

While a mere question of degree is not a patentable thing under our law, where the question of degree develops a large commercial practice, even though the difference in results is hard to define, there is an obvious discovery in the art or large commercial practice would not follow.

It appears a very just decision that there was invention under U. S. Patent No. 835,120, and that some degree of range in practice must be adopted to define infringement. One per cent was given as a convenient gross amount of oil.

If industry has now discovered that more than 1 per cent of oil is equally useful, it is their right to use such degree, but this cannot in any way reflect upon the opinion of the court, because this knowledge was discovered long after, and as a result of long experience with less than 1 per cent of oil, which gave birth to the flotation industry.

As to who was the true inventor of less than 1 per cent of oil is not now at issue, as Patent No. 835,120 has the legal status. The courts found justly that a large commercial practice followed at once the discovery that less than 1 per cent of oil was practical and sustained the patent that described such.

The purpose of the Patent Office is to encourage invention by offering reward to inventors in the form of royalty protection, and thus create advance for the community.

The result has for many years been very disastrous to inventors who are not capitalists and have no time for business practice.

Industry made the discovery that it could employ counsel and cause court delays and so defeat the just reward of invention, and that such practice was more economical than paying royalties. This is in direct defiance of the patent laws which grant a period of

royalty protection, because the refusal to pay royalty discourages invention and delays community advance.

In the end the present practice of industry is a short-sighted one as a community loss is the greatest that can occur.

The flotation decision is a most valuable precedent, and probably establishes the fact that invention that brings commercial practice will be protected, irrespective of academic theory or engineering practice that become obvious after the patent was issued.

How much better it would be for industry to aid the inventor, where they desire to use his invention or supposed invention, and advance him money to test the validity if in question, and thus prevent capitalizing of patents solely for monopolistic purpose.

The practice of attempting to own the inventor or force him to assign at a nominal price, or before there is any established value, should be deplored as a form of piracy.

Invention or research is a vital force that precedes all industry advance, and while radically different in mentality is in our advanced civilization perhaps quite as important. Even though the ownership of the flotation patents is to be deplored, if the lesson is learned by industry to deal with the inventor directly and permit him to hold and administer his inventions, aiding him in so doing, it is cheap even at the price of \$50,000,000 more or less that it may cost industry to settle its bills.

The inventor should profit only by the commercial value and validity of his invention through royalty. Industry should be glad to pay royalty in commercial degree as community progress, and profit solely by manufacturing and salesmanship.

There should be no such thing as control of common commodity patents by industry.

The interpretation of the law by the courts is practically always an echo of public opinion of the same kind, and "A. B. C." will do well to look a little beyond the argument he presents.

METALLURGICAL ENGINEER.

### Electrolytic Oxidation of Sulfurous Acid

*To the Editor of Metallurgical & Chemical Engineering*

Sir:—I was much interested in the article by Messrs. M. De Kay Thompson and N. J. Thompson on "The Electrolytic Oxidation of Sulfurous Acid," page 677 of your issue of Dec. 15, 1916. This subject has been of no small importance in the field of copper leaching investigation where the copper is obtained by the electrolysis of a sulfate solution and the possibilities of making acid and lowering cell voltage by the utilization of such an anode reaction become worthy of attention. Of course, the same subject is intimately connected with several important lines other than copper leaching.

It would be interesting if Messrs. Thompson would give us figures and a discussion on the possible application of their data to a commercial cell. In the latter case, of course, the anode would not be platinum and the reaction could not be so perfect, but an idea would be obtained of the maximum amount of sulfur dioxide per commercial unit that could be possibly oxidized under given conditions.

Multiplying current density by current efficiency by the factor 1.192 (which is the grams sulfur dioxide



oxidized per ampere-hour when this is the sole reaction) gives at once the weight in grams of sulfur dioxide completely oxidized to sulfuric acid per square decimeter of anode surface per hour, for any given experiment. The resulting figure is a measure of the absorption yield of sulfur dioxide or the production yield of sulfuric acid or the relative velocity of the desired anode reaction, according to the view-point. It could be applied at once to any commercial cell unit of given size. The shape of the curve would evidently be the same as that of the current efficiency curves as given by Messrs. Thompson.

The current efficiencies obtained in the investigation seem remarkably good. Under such conditions the depolarizing effect would seem to be at its maximum and indeed in the case of 10 per cent sulfuric acid no anode oxygen was evolved at all even with very considerable current densities. At first glance, it might seem that the anomalous condition prevailed of perfect absorption with no depolarizing effect, as the cell voltage was 4.00 for 10 per cent sulfuric acid and a current density of 19 amperes per square decimeter (the presence of the porous cup diaphragm and the extra resistance thereby introduced into the circuit is kept in mind). However, 19 amperes per square decimeter is 176 amperes per square foot, an extremely high density when compared with metal deposition practice, where current densities commonly range between 10 and 40 amperes per square foot. Any depolarizing effect is therefore masked by the high electrolytic resistance drop and Messrs. Thompson's figures on cell voltage can hardly be discussed without further information from them. Comment on any depolarization observed and on this phase of the subject in general would be extremely interesting.

Apropos of a sulfur dioxide anode, several years ago the writer directed an investigation of a sulfur dioxide-platinum black anode immersed in a solution of copper sulfate, sulfurous and sulfuric acids. On open circuit the single potential was found to be + 0.42 volts (electrode positive to the solution; here as elsewhere the sign refers to the charge on the electrode). A normal calomel electrode assumed to be + 0.56 volts was used in making the measurements. A copper cathode in the same solution had a single potential of + 0.54. The open circuit combined cell voltage was therefore only  $0.54 - 0.42$ , or 0.12 volts, a figure that looked extremely promising.

Sad to relate, on applying something like a commercial current density to the cell, about 30 amperes per square foot at the anode and 15 at the cathode, the sulfur dioxide electrode "broke down" completely, even changing its sign, becoming -1.10 volts. The copper cathode went to + 0.70, so that the cell voltage at the electrodes was  $1.10 + 0.70$ , or 1.80 volts. The electrodes were spaced 1.5 in. between surfaces and with the strength of solution used the total electrolytic resistance drop amounted to 0.55 volts. The external cell voltage under running conditions was therefore  $1.80 + 0.55$ , or 2.35 volts total, instead of the approximately 0.7 volts that we had hoped for. Some sulfurous acid was oxidized to sulfuric but the reaction seemed too slow to appreciably depolarize the cell or to promise any practical advantages. That using still higher current densities might have caused any improvement in the matter did not occur to anyone at the time and the investigation was therefore dropped as leading to negative results. It was made with some care and using delicate instruments, but not under exactly research conditions or using pure materials.

The writer has ever after been somewhat skeptical of the commercial utility of using sulfur dioxide as an anode depolarizer when electrolyzing copper sulfate solu-

tions with insoluble anodes. However, some very broad claims have been made in the past, even down to running cells with no more energy consumption than in the case of soluble anodes. Of course catalytic agents in the solution, iron, etc., have a large effect on the oxidation of sulfur dioxide at the anode and would help to give the result desired. Still, it would be interesting to learn of a single running plant at the present time where depolarization with sulfur dioxide is being satisfactorily used on a commercial scale.

MAURICE R. THOMPSON.

General Electric Co.,  
Power and Mining Department,  
Schenectady, N. Y.

\* \* \*

*To the Editor of Metallurgical & Chemical Engineering*

SIR:—Replying to Mr. M. R. Thompson's comments on the article by Mr. N. J. Thompson and myself, I would say that the high voltage found necessary to get the currents desired was due partly to the diaphragm and to the polarization of the hydrogen liberated. On account of lack of time, no single potential measurements of the anode were made. These, of course, are important and it is hoped to add them later. It would appear from the measurements of your correspondent that there is a resistance to the oxidation of sulfurous acid similar to that observed by Haber and Russ in the case of hydrochinon (*Z.f. phys. Chem.* 47, 257, 1904).

M. DEKAY THOMPSON.

Massachusetts Institute of Technology,  
Cambridge, Mass.

### More Publicity Urged on Industrial Research Laboratories

*To the Editor of Metallurgical & Chemical Engineering*

SIR:—The undersigned Committee on Engineering of the General Committee on Research of the American Association for the Advancement of Science feels that it is timely to issue the following appeal to the industrial research laboratories of the country.

In the course of work done in the numerous industrial laboratories of America, many physical and commercial constants and data of great scientific interest and value are doubtless arrived at, and which may, for a certain period of time, constitute an asset of considerable commercial value to the particular corporations in question. During this period, everyone recognizes the proprietary right of the industrial laboratories to the retention of this information.

A time frequently arrives, however, when such scientific information loses its commercial value (often by being duplicated in other laboratories), and just at this point we wish to impress upon the industries their obligation to enrich scientific literature with such facts and data, which might otherwise be lost or forgotten.

Some of our industries have been reproached with the suspicion of acting as sponges, in that they absorb an immense amount of useful information from scientific literature without giving any return in kind. This suspicion would be entirely removed if, from time to time, scientific information which has ceased to be of commercial value were contributed by them to its appropriate channel and thus became available to all scientific workers throughout the world.

If any doubt exists as to the appropriate channel for the publication of such scientific data and communications, the secretary of the A. A. A. S., Dr. J. McKeen Cattell, Garrison-on-Hudson, New York, will be glad to act as intermediary and to forward such communications to the proper scientific body.

A. E. KENNELLY, J. W. RICHARDS, A. SAUVEUR, A. N. TALBOT, C. C. THOMAS.



### Who?

From the "Transactions" of the Buffalo Section, Institute of Literary Engineers.

Who takes the pleasure out of life and makes existence Hell?

Who fires a real good-looking one because she cannot spell?

Who substitutes a dictaphone for coral-tinted ear?

The penny-chasing, dollar-wasting efficiency engineer!

### Coming Meetings and Events

American Institute of Mining Engineers, annual meeting, New York, Feb. 19-22, 1917.

American Chemical Society, New York Section, Nichols Medal Award, Chemists' Club, New York, March 9, 1917.

American Chemical Society, spring meeting, Kansas City, Mo., week of April 9, 1917.

American Electrochemical Society, spring meeting, Detroit, Mich., May 2-5, 1917.

American Electrochemical Society, autumn meeting, Pittsburgh, Oct. 3-6, 1917.

### National Safety Council Broadening Its Activities

The National Safety Council, the leading accident prevention agency in the country, is broadening its activities, and has commenced monthly publication of a series of safe practices leaflets. The council has a committee of fifty safety experts working out the maximum and minimum requirements in safeguarding, and has already issued three leaflets on this important work. These are entitled respectively "Ladders," "Stairs and Stairways" and "Boiler Rooms." Others will be issued monthly.

The purpose and scope of these leaflets is explained in a preliminary announcement in the leaflet on "Ladders" as follows:

"The National Safety Council was established to help free mankind from accidents, and, to that end, to gather to itself as members all those concerned and interested in the safety movement, and to bring about an understanding of accident causes and find and apply remedial measures.

"The field has been new and the various services rendered have met the need, in so far as the council's modest income from its nominal fees, its experience and the experience of its members have permitted. The time has now come when the pioneering period has partially passed and our accumulated experiences permit, and the awakening needs require, that the great amount of collected and obtainable safety information be put into concrete and popular form for the benefit of the cause.

"The executive committee has, therefore, decided to compile the best information obtainable about safe practices of all kinds and to present same in a logical and orderly form. It is hoped it will become an encyclopedia of information on all phases of the subject. It is fully realized that this is a very large undertaking, but this need not deter us if the accomplishment is necessary to the progress of the council's purpose and its service to its members. The council will simply do the best it can with such a large and important subject, relying upon the co-operation of the members for the success of the plan.

"The gathering and compiling of data will have to be sectionalized to cover the field."

The organization includes in its membership many of the leading industrial concerns of the country, and

has offices in the Continental & Commercial Bank Building, Chicago. Mr. W. H. Cameron is general manager.

### Award of the Elliott Cresson Medal to E. F. Northrup

The Franklin Institute has recently awarded its Elliott Cresson Gold Medal to Edwin Fitch Northrup, Ph.D., research physicist, of Princeton, N. J.

This award was made in recognition of a special type of electric furnace developed by Dr. Northrup, in which a temperature of more than 3000 deg. C. can be developed, and of his pyrometric methods and new pyrometric apparatus for the direct and accurate reading of high temperatures up to 1600 or 1700 deg. C. Means are provided whereby, with a slight modification in the pyrometric apparatus, rapid measurements can be made of the resistivities of many molten metals and other liquid materials through a wide range of temperature.

### The Western Metallurgical Field Shale Oil

**Experimental Plant for the Extraction of Oil from Shale for Flotation.**—After a lengthy and careful investigation as to the suitability of oils extracted from Colorado shale for flotation, the Oil Shale Mining Company of Colorado has decided to install an experimental plant for the extraction of the oil near De Beque, Mesa County, Colorado. Endeavors are being

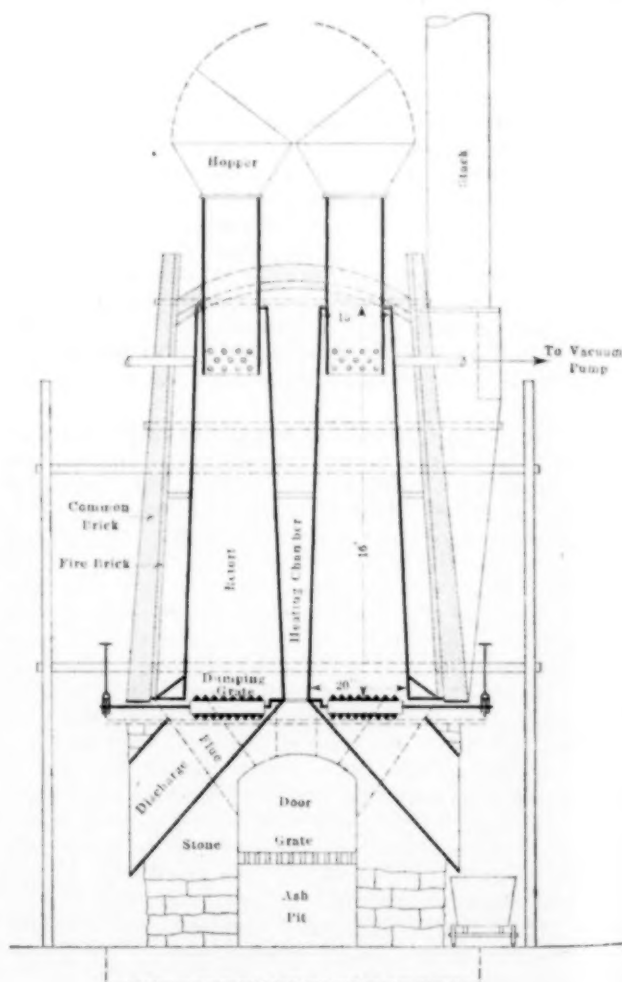


FIG. 1—RETORTS USED IN THE DISTILLATION OF OIL FROM SHALE

made to have the necessary apparatus ready for shipment by the beginning of March.

The initial plant unit will consist of six retorts, similar to the Henderson type, in successful use in Scotland, where the shale oil industry has been in operation for more than fifty years. The retorts are made of cast iron and are 16 ft. in height, tapering from 20 in. at the bottom to 16 in. at the top. At the bottom of each retort is a grate, through which the cinders are removed to a discharge. Near the upper end of each retort is an exhaust, connected with a low vacuum pump, which draws off the volatile vapors which are to be condensed. The retorts are charged from the top through hoppers, which are kept closed during operations. The heating is done from the outside. A grate at the bottom of the chambers surrounding the retorts supports the fuel bed. The products of combustion impart their heat to the retorts and the charge, and then go to the stack. The heating chamber is made of common brick lined on the inside with a layer of firebrick. The retorts are placed in two rows of three each. Fig. 1 shows a vertical section through the apparatus.

The shale, suitably crushed, is fed through the hoppers into the retorts and subjected to distillation under diminished pressure. The vapors are withdrawn through the exhaust near the top of the retort, and are condensed by passing through a metal coil immersed in cold water. No efforts are being made at the present time to fractionate the oil, as the demand of the product for flotation processes is such that the company is looking toward the money made by these sales to defray the expenses of any further experimental work.

It is claimed that the oil thus obtained is a good frothing agent, and does not become as viscid as coal-tar oils when used in connection with cool water under identical conditions. It is estimated that the unit as constructed will yield 20 bbl. of oil per day at 42 gal. to the barrel. The market value per barrel will range from \$4.50 to \$6. A ready market for the output is claimed by the company.

#### Company Reports

**Annual Report of The Granby Consolidated Mining, Smelting and Power Company, Ltd., Ending June 30, 1916.**—During the fiscal year the production of the various plants connected with the company from 1,897,251 tons of ore treated was 42,198,083 lb. of copper, 487,845 oz. of silver and 44,848 oz. of gold. At the Anyox Smelter, Anyox, B. C., three furnaces were operated on green ore, while a fourth was used for regrading matte. The tons of ore per furnace day increased from 630 to 692 and the tons of charge from 846 to 929. The cost of smelting and converting was \$1,804 per ton of ore, or \$0.073 less than for the previous year. The amount of ore treated at this plant was 822,919 tons, yielding 29,562,177 lb. of copper. The Anaconda charging system was installed during the course of the year, and the shaft depth of No. 4 furnace was increased by 5 ft. for experimental purposes. The latter change proved so satisfactory that any new furnaces will be built on the same type.

The Grand Forks Smelter handles approximately 500,000 tons of low-grade copper material high in silica. Eight furnaces were in blast. Up to the present time this material was not taken in the ore reserves, but on account of the high price of copper which prevailed during the past year the handling of this material became profitable. For the first six months of the year the cost of smelting and converting was \$1.233 per ton. The plant also converted 21,428 tons of matte from the Anyox smelter.

### Philadelphia Hearing in Miami Flotation Suit Before Court of Appeals

The hearing of the Minerals Separation vs. Miami Copper Company infringement suit commenced before the United States Court of Appeals for the Third Circuit in Philadelphia on Tuesday, Jan. 30, and continued until the following Saturday noon.

In the lower court, the United States District Court for the District of Delaware, Judge Bradford had rendered the opinion that the principal patent, 835,120, for a fraction of 1 per cent of oil in its principal claims, was valid and infringed; also that the patent 962,678, for soluble frothing agents was, in the claims under consideration, valid and infringed, but that the third patent in suit, 1,099,699, for aromatic hydroxyl frothing agents without acid and in the cold, was invalid (*Met. & Chem. Eng'g*, Oct. 15, 1916, Vol. XV., pages 433 and 441).

The appeal was therefore a cross appeal; the Minerals Separation appealed from Judge Bradford's finding as to the third patent in suit, while the defendant in the lower court, the Miami Copper Company, appealed on the other hand as to the findings on the first and second patents in suit.

Judge Bradford in his opinion had stated that the reduction of oil in patent 835,120 was so great as compared with the amount of oil used in prior-art processes as in itself to constitute invention—that is, that the reduction of oil *per se* was patentable. This was at variance with the law finding of the Court of Appeals in San Francisco which had maintained in the Hyde case that the mere reduction of oil was not patentable and had found upon matters of fact that there being only a difference of oil, patent 835,120, was invalid.

In the Supreme Court of the United States the patent 835,120 was found to be valid, this decision following Judge Bradford's by only a few weeks.

This condition then left the case before the Circuit Court of Appeals in the Third Circuit (the Miami case) with two main contentions.

There was a difference of opinion as to what the interpretation of the Supreme Court decision really should be.

The Miami attorneys claimed that the Supreme Court had in its decision affirming the validity of the patent definitely limited the patent to a fraction of 1 per cent of "oil" in combination with a novel agitation, greater than previously used, by "beating in" air, and with the formation of a characteristic "froth," different from any heretofore produced.

Under this construction the decision of the United States Supreme Court was at variance with that of Judge Bradford and in harmony with the Court of Appeals of the Ninth Circuit, as to law, but at variance with the Ninth Circuit as to fact.

The pith of the question before the court as to infringement or otherwise therefore, was largely dependent upon the manner of interpretation of the Supreme Court decision in the Hyde case. In the discussion and for illumination of this, various prior-art processes were considered and experiments shown.

Preliminary to the arguments the court requested each side to present "a skeleton" of their respective cases, so that the court might then more understandingly follow the arguments.

After this each claim was taken up separately and the Miami attorneys stated what part of each claim they acknowledged as used in the operation of their process and what part they denied. For example, "ore pulp"—"admitted"; "small quantity of oil, fraction of 1 per cent"—"admitted"; "agitation to produce a froth"—



"denied"; "and remove the froth"—"denied," etc. Thus through each claim was analyzed.

The judges sitting were Judges Buffington, MacPherson and Wolley. The argument on behalf of Minerals Separation was made by Mr. Henry D. Williams and the closing by Mr. Wm. H. Kenyon; for the Miami Copper Company the argument was made by Mr. Louis Marshall and Mr. W. A. Scott, and the closing by Judge George Gray of Wilmington, Del. The latter had previously sat upon the very bench he was addressing, and with two of the judges now on the bench, Judges Buffington and MacPherson.

As in the court demonstrations in Wilmington, illustrative of the prior art, there was a wide discrepancy as to the interpretation of the disclosures. (See *Metallurgical and Chemical Engineering*, July, 1915, Vol. XIII, page 409.)

There was also an important difference in this court in the experimental illustration of the Miami Copper Company's process.

The Minerals Separation experts agitating the pulp violently in "Gabbetts," which they stated represented a centrifugal pump and Pachuca tank in the Miami initial plant, whereas the Miami experts very gently mixed the pulp and oil and then introduced to the canvass-bottom cell and the spiral coil apparatus of prior patent 793,808, which process Judge Bradford had stated in his decision was the process of the Miami Copper Company. The Minerals Separation experts obtained their air for the cell from compressed-air cylinders at 200 or 300-lb. pressure, admitted to the cell through reducing-pressure valves, which psychologically at least impressed the court with the idea that the air was "shot into" the pulp—an impression which seemed to be dispelled when the court witnessed the Miami company's experimental illustration of their process.

Judge Gray made the closing argument for the Miami company. He dwelt upon the limiting conditions imposed by the United States Supreme Court as to novel violence of agitation and characteristic permanence of froth, contending that to broaden the patent 835,120, so as to cover the operations of the Miami Copper Company's process would in effect be to reincorporate the original claim 12, which the U. S. Patent Office had denied, and which patentees had withdrawn, a claim in which the terms "agitate to produce a froth" had not been used.

Mr. Kenyon made the closing argument for the Minerals Separation, contending that the apparent limiting clauses in the Supreme Court's decision were part of the description, not of the decree, and that the "short step" referred to was the step "turning failure into success," and that the patent was, therefore, in effect "a master patent" if not actually the pioneer patent.

### Chemical and Electrical Porcelain

The "Chemical and Electrical Porcelain" meeting held jointly by the New York Section of the American Electrochemical Society, American Chemical Society and Society of Chemical Industry on Friday evening, Feb. 9, was very well attended and the papers were very interesting. Dr. COLIN G. FINK presided as chairman of the New York Section of the American Electrochemical Society which had made the arrangements for the meeting. The international situation has made us dependent on domestic sources for porcelain and, as in many other lines, the manufacture of this commodity is commanding a great deal of attention. This is especially true for chemical porcelain for which we were formerly dependent on foreign sources of supply.

Dr. CHARLES F. BINNS, of Alfred, N. Y., director of the New York State School of Clay Working and Ceramics, at Alfred, gave an interesting illustrated talk on the history, constitution and manufacture of porcelain, showing some microphotographs which brought out many interesting points. The various furnaces and other apparatus used were also described. Dr. Binns has devoted a life-time to the subject of ceramics and did not think the volume of American business in chemical porcelain would warrant a separate factory for its manufacture, although we have the raw materials and everything necessary for its manufacture, if the proper technique is observed. He also thought that protection was necessary to protect the industry from foreign competition after the war.

Mr. L. E. BARRINGER, president of the American Ceramic Society and chief engineer of insulation of the General Electric Company, gave an interesting account of the manufacture and properties of porcelain for electrical insulation, supplementing his remarks with lantern slides. He showed several pieces of porcelain ware made by the dry process for low voltages and by the wet process for high voltages. One of the interesting features of his talk was the description of a continuous kiln fired in the center with the material going in at one end, gradually approaching the hot center zone and being drawn out at the cool end. This kiln saves 50 to 60 per cent of the heat used in an intermittent kiln.

Dr. A. V. BLEININGER, of the Bureau of Standards, Pittsburgh, Pa., took up a discussion of the physical and chemical characteristics of porcelain, explaining the effect of the various constituents on the final product. The Bureau of Standards has done a great deal of work in the study of porcelain, which is of great value to our manufacturers.

In the discussion Dr. W. D. Bancroft, of Cornell University, suggested that straight sillimenite would be an interesting product to study, not so much from commercial considerations but from a theoretical standpoint. Dr. Bleininger said the potential difficulties were very great in producing this. Mr. R. C. Schroth, Jr., president of the Laboratory Supply Company, Columbus, Ohio, agents for the Ohio Pottery Company, said this company had conducted considerable research in the last two years and that they were firing at the high temperatures used abroad. Several samples of this company's chemical porcelain were on exhibit.

### Methyl Alcohol and Acetone as By-Products of the Soda Pulp Industry\*

By Alfred H. White and John D. Rue

The introduction of lime as an agent for the disintegration of straw for paper-making is attributed to the Chinese, and the process was in use in Europe in the seventeenth century. The use of caustic soda under pressure for the manufacture of pulp from straw was introduced by M. A. C. Mellier, of Paris, who took out a French patent in 1854 and United States patent 17,387 in 1857. The real development of the woodpulp industry came in America, although based on patents of Watt and Burgess, of London, who in 1853 took out an English patent, and in 1854 an American patent, 11,343, whose rights were assigned to Messrs. Ladd and Keen, of New York.

The first plants for the operation of their process seem to have been erected at Royers Ford, Pa., and later at Manayunk, near Philadelphia, by the American Wood Paper Company.

\*Read at the annual meeting of the Technical Association of the Pulp and Paper Industry, New York, February 7, 1917.



The American practice early differentiated itself from European by the use of more concentrated caustic liquors, so that the recovery of the soda by evaporation of the black liquors and incineration of the residue was a feature of the process almost from its infancy in this country. The attempts made in Germany and Scandinavia to cook with weak liquors which might be wasted were not commercially successful, and the American system was introduced into Europe soon after 1870. After its introduction the Germans made important improvements in methods of washing the pulp, concentrating the black liquor and calcining the soda.

The system of alkali recovery in the early mills was copied somewhat from the Leblanc soda process. The liquor was concentrated in a series of pans heated by direct fire, and the liquor passed from pan to pan, approaching nearer the fire each time. The thick mass was finally calcined in an open pan. Attempts to still further utilize the heat of the gases from the calcining furnace led to the construction of towers through which the dilute black liquor fell in a spray or tumbled down over a series of shelves against the flow of the hot gases.

The introduction of the multiple effect evaporator marked an important step in the industry. The evaporator was what is now the well known Yaryan type, invented by Homer T. Yaryan of Toledo, Ohio, in 1886. It was developed and adapted to the concentration of waste liquors from the soda mills by Augustus G. Paine of New York, who in 1890 consolidated several mills under the name of the New York and Pennsylvania Company. To this same company is due the introduction of the rotary kilns for burning the concentrated black liquor. A patent on this feature taken out by L. D. Armstrong of the company in 1892 (U. S. Patent 480,702), and patents were also taken out by S. D. Warren & Co. of Boston, Mass. The commercial problem was worked out by the two companies in collaboration, and the type of rotary then developed is still in general use in soda pulp mills. The organic material in the concentrated liquor is nearly sufficient to complete the evaporation and maintain the required amount of combustion. Some of the waste heat is used to generate steam in boilers set into the flues. No attempt is made in the present process to recover any valuable product but the alkali.

Our own interest in this subject has been in trying to recover methyl alcohol and acetone by destructive distillation of the black liquors. We have been assisted in this both in the laboratory study and in the tests on the larger scale by the New York and Pennsylvania Company, to whom we wish to express our thanks. Our acknowledgments are especially due to Mr. George K. Spence and Mr. John Krauss, chemists at the Clarion mill, for their unflinching courtesy and assistance.

#### THEORETICAL

The process of destructive distillation of wood to produce methyl alcohol, acetic acid and charcoal is an old one. The question as to the constituents of the wood which produced these products has, however, had little study. Klason<sup>1</sup> has pointed out that the alcohol must come from the lignin, since the destructive distillation of cellulose gave no alcohol in his tests. We have in the main confirmed his results since the destruction distillation of soda pulp, which is not entirely free from lignin, gave in our tests only 0.06 per cent by weight of methyl alcohol, while hardwood pulp chips gave 1.47 per cent by weight. It is therefore evident that the material of the wood which produced the alcohol went into solution in the soda digester or was decomposed entirely and evolved as gas.

Indirect evidence is afforded by the tests of Bergström and Fagerland<sup>2</sup>, who determined the amount of methyl alcohol evolved with the gases given off during a sulphate cook and found that 1.3 per cent on the weight of the pulp, or perhaps 0.65 per cent on the weight of the wood, was converted into alcohol during the cooking process.

The total methoxy groups in the wood as determined by digestion with strong hydriodic acid is frequently assumed to give the maximum amount of methyl alcohol which can be obtained from a given substance. Benedikt and Bamberger<sup>3</sup> tested wood in this way and obtained indicated maximum yields of methyl alcohol varying from 4.38 to 6.52 per cent by weight of the wood, according to the species. This figure is so large that it is evident that there must still be possibilities for considerable alcohol recovery from the waste liquors. Streeb<sup>4</sup> showed that the methoxy content of the lignin acids precipitated from black liquors by mineral acids was between 5.78 and 5.98 per cent. Attempts were made by H. Lowe in 1861 and Tessie du Motay in 1871 to precipitate the organic matter by carbon dioxide to facilitate the recovery of the waste lyes, but their results were not commercially successful. Rinman<sup>5</sup> has patented a process similar to this involving precipitation of the humus matter by carbon dioxide or stack gases and destructive distillation of the solid residues. Our own experiments have shown that lignin from hard wood precipitated in this way gives on destructive distillation 1.62 to 2.20 per cent by weight combined alcohol and acetone, which is only about two-thirds as much as an equivalent amount of highly concentrated black liquor gives when distilled under the same circumstances.

#### LABORATORY TESTS

Attempts to distill this concentrated black liquor by heating the full charge in a still such as might be used for tar gave much trouble. The liquor foamed badly and after the destructive distillation was started an exothermic reaction developed which caused a violent evolution of gases. The process of feeding the liquor gradually into an inclined preheated retort gave much better results. In this way successive films of the liquor were subjected to distillation at an even temperature. Not only could the temperature be controlled, but also the length of time which the vapors remained in the hot retort and the extent of the contact with the charcoal, which latter agent has been shown to have a powerful catalytic effect. Experiments showed that the best results were obtained at temperatures even lower than those usual in wood distillation, provided an adequate time was allowed. The temperature of 550 to 600 deg. Fahr. (288 to 316 deg. C.) seemed most desirable.

The laboratory retort used for these tests was made from a piece of gas pipe 3 in. in diameter and 18 in. long which was set in an inclined position. The liquor entered continuously through a side arm at the middle of the retort and the gases left at the upper end. The results of a series of seven consecutive tests with this retort on liquor averaging 41.3 deg. B. are given in Table I. The average result of the seven tests shows 3.81 per cent by volume of methyl alcohol and 0.56 per cent by volume of acetone in the aqueous distillate. Five of the tests are on liquors from hard wood, mostly beech, and two are from the soft wood, mainly bass wood. It is noteworthy that this soft wood which, if distilled directly, would yield very little alcohol, gives fully as good results after the soda treatment as does the hard wood. Other tests on poplar and jackpine liquors have shown

<sup>1</sup>Papier-Fabrikant, 7, 27-32, 78-82, 104-6, 129-31 (1909); 8, 970-1.

<sup>2</sup>Monatshefte, 11, 260-7, 1890.

<sup>3</sup>Dissertation Göttingen, 1892.

<sup>4</sup>Papier-Fabrikant, 10, 39-41, 101-4, 1912; 12, 212-5, 1914.

<sup>5</sup>Ztr. f. angewandte Chem., 23, 1252-7, 1910.

good yields of methyl alcohol, though the conditions necessary for the best results have not been worked out.

In this series of Table I the rate of feeding was such that the vapors stayed in the retort from 15 to 30 seconds. Under these circumstances the liquor as it entered the retort foamed and was charred almost immediately, so that the whole interior space of the retort became speedily filled with a very porous black ash which offered a very large amount of contact surface. In some tests the upper part of the retort was filled prior to the test with a wire basket containing black ash, to give more contact surface. This did not seem to be neces-

TABLE I—LABORATORY DISTILLATIONS OF BLACK LIQUOR FROM SODA PROCESS IN RETORT MADE FROM 3-INCH IRON PIPE

Test No.	Specific Gravity of Liquor in Degrees Be	Kind of Wood	Volume Per Cent of Aqueous Distillate	COMPOSITION OF AQUEOUS DISTILLATE, PER CENT BY VOLUME		Average Retort Temperature, Degrees F.
				Methyl Alcohol	Acetone	
5	40.4	Hard	66.0	4.03	0.45	513
6	40.4	Hard	61.0	3.64	0.48	548
7	40.4	Hard	67.0	3.48	0.67	506
8	41.0	Hard	65.4	3.47	0.69	573
9	41.0	Hard	67.5	3.73	0.53	567
10	43.0	Soft	67.6	4.27	0.67	567
11	43.0	Soft	64.0	4.06	0.66	615
Aver.	41.3		65.5	3.81	0.56	

sary at the higher temperatures but did give better results at the lower temperatures. Test Number 3, of this series was distilled at a temperature of 555 deg. Fahr., which was entirely satisfactory in other cases, but in this test the liquor was fed at a rapid rate so that the gases stayed in the retort only eight seconds, as against the fifteen or thirty seconds of the other tests. In this test the destructive distillation was complete, as shown by the well charred black ash, but the combination of low temperature and short time showed its effect in the large volume of distillate, 76.0 per cent by volume, and the low yield of alcohol and acetone, which together amounted to only 1.96 per cent by volume of the distillate, whereas the average of the other tests gave 4.37 per cent.

#### LARGE SCALE TESTS

It was evident from the laboratory tests that in order to obtain good yields of alcohol and acetone it was advisable to conduct the distillation rather slowly and at a temperature well below a red heat and not necessarily above 550 deg. Fahr. This temperature is no higher than that of the waste gases from many steam boilers and made it evident that steel could be safely used as the retort material. The greatest practical difficulty in transferring the process to a large scale came from the rubbery nature of the product during the initial stages of the treatment. Starting as a thick syrup at 40 to 42 deg. B., it changes as the water is driven off to a mass which is sticky and plastic while hot and tough and hard while cold. It also foams strongly during the first stages of the destructive distillation.

The two previous experimenters who have recorded their work in this field have both emphasized the difficulties in handling this material. Meyer<sup>4</sup> proposes to absorb the thick syrup in briquettes of charcoal previous to the distillation, and Sandberg<sup>5</sup> uses a combination of mechanical scrapers and screw conveyors to work the material through the retorts. The authors have found it preferable to allow the liquid to flow as a film over the inside of the retort and build up an increasing thickness of charcoal, and then discharge the retort altogether and start afresh.

<sup>4</sup>U. S. P. 407, 442 July 23, 1889.  
<sup>5</sup>British patent 23,125 of 1912.

Both vertical and inclined retorts have been tested. The vertical retort was rectangular, 12 by 18 in. in cross section and 10 ft. in height above the grate bars. The top was closed by a lid in a liquor seal that also served as the means of introducing the liquor which trickled over the edge of the retort and ran down the sides, charring as it went. It was found, however, that the liquor ran down the hot sides of the retort so rapidly that it reached the bottom while still liquid and collected in a pool at the bottom of the retort. The catalytic effect of the carbon was also much less in this large retort than in the laboratory retort, so that the best results were obtained when the vapors stayed in the retort 75 seconds instead of the 15 to 30 seconds necessary with the smaller retort. Even under these conditions the yields were only about 60 per cent as much as those with the smaller retort.

The next attempt was to use an inclined retort which was merely the laboratory retort on an enlarged scale. After preliminary tests to determine the proper angle a battery of twenty-four retorts was constructed. Each retort was made from steel well casing 9.6 in. in diameter and 12 ft. long set with the upper end 3 ft. higher than the lower. This bench was operated twenty-four hours a day for three weeks without any serious operating troubles. The black ash was well charred and leached to a colorless solution, leaving a soft and porous charcoal. There was absolutely no volatilization of soda ash from the retorts, showing that the loss of soda from the rotaries could be entirely eliminated. The doors of the retorts were not properly made and could not be closed tightly. If suction was kept on the retort air leaked in and destroyed some of the products. If there was back pressure on the retorts the gases leaked out and some of the distillate was lost. An inadequate condenser also caused loss. Because of these defective details the average results were lower than they should have been.

For short periods of time as good results could be obtained as on the laboratory scale but, as the average of a day's run, the strongest distillate obtained was when 439 gal. of 40.1 deg. B. liquor from bass wood were distilled and gave a distillate containing 2.75 per cent by volume of methyl alcohol and 0.46 per cent acetone, which is about 75 per cent of the strength obtained on the laboratory scale. There does not seem any reason to doubt that, with leaks eliminated, this apparatus could give results in every way as good as those obtained in the laboratory. The capacity of this plant was 1 gal. of concentrated liquor per retort per hour or 576 gal. per day of twenty-four hours for the unit installed. It was an experimental plant with the feeding of the liquor and discharge of the black ash controlled by hand, which made the labor cost altogether too high for commercial operation. The chief problem still remaining is that of designing efficient machines for replacing hand labor in these operations.

In this particular installation natural gas was burned, but tests for several successive days showed that the black ash as it came from the retort could be completely burned on grates below the retorts, fused white soda ash dripping through the grate and collecting as stalagmites in the pit below. This procedure is not, however, wholly advantageous, since there is loss of soda mechanically carried off with the smoke gases just as there is from the rotaries. Better practice would involve leaching the black ash as usual and then burning the leached wet carbon in gas producers or, after drying by waste heat, burning it directly on grates or in the form of powdered fuel.

The yields to be obtained from the destructive distillation are shown in Table II, calculated to a basis of 1000 gal. of 40 deg. liquor and also to a basis of a cord of



wood. The figures given are really for hard wood, but the yields from soft wood per 1000 gal. of black liquor are practically the same and the yields per cord of soft wood need to be corrected only for the difference in the weight per cord to make them approximately accurate. The yields of methyl alcohol and acetone are all based on analyses of the distillates, since no refining tests on the large scale have been made. No difficulties in the refining process, however, have been indicated by the

TABLE II—YIELDS FROM DESTRUCTIVE DISTILLATION OF CONCENTRATED BLACK LIQUORS FROM SODA PULP PROCESS

Composition of black liquors of 10° Bé:			
	Percentage by Weight	Pounds per 1,000 Gals.	
Water	31.7	3,640	
Organic matter	10.0	4,600	
Ash	28.3	3,260	
	100.0	11,500	

Yields from 1,000 gals. Black Liquor of 40° Bé:			
	Percentage by Weight	Pounds	Gallons
Aqueous distillate	41.6	4,790	575
Tar	5.6	650	71
Black ash	44.5	5,120	.....
Gas (by diff.)	8.3	940	.....
	100.0	11,500	.....

Carbon after leaching Black Ash 2100

Black Ash Composition:		
	From Hard Wood, Per Cent	From Soft Wood, Per Cent
Combustible	41.3	44.3
Ash	58.7	55.7

Aqueous Distillate:			
	Percentage Composition	YIELD FROM 1,000 GALLONS 40° LIQUOR	
		Pounds	Gallons
Methyl alcohol	3.81 by vol.	145.0	21.9
Acetone	0.56 by vol.	21.6	3.2
Ammonia (as NH <sub>3</sub> )	0.04 by wt.	4.6	.....

## Yields per Cord of Wood put into Digester:

Based on 890 pounds of soda ash per cord of wood weighing when chipped 1,100 pounds.  
 890 pounds of soda ash are found in 273 gallons of 40° liquor.  
 273 gals. 40° liquor yield 157 gals. aqueous distillate, containing:  
 { 6.0 gals. methyl alcohol 100 per cent.  
 { .9 gals. acetone  
 { 6.9 gals. alcohol and acetone.

laboratory distillations. The distillate contains no acid and therefore iron condensers and stills may everywhere replace the more expensive copper. Neither is there any need of the expensive first distillation of the entire liquor which the wood distillers must make to separate the acetic acid from the tar. The liquor as it comes from the tar separating tanks is practically in the same stage as that from the wood distillation after the first distillation has been completed and the acetic acid has been neutralized.

## TAR

The tar forms about 11 per cent by volume of the total distillate from hard wood. It has a specific gravity of 1.10 and a pungent odor entirely different from that of either wood tar or coal tar. On distillation, according to the method of Church<sup>4</sup>, it gave the following results:

<sup>4</sup>Jour. Ind. and Eng. Chem. 3, 227-233, 1911; 5, 195-6, 1913.

	Per Cent
Water	8.6
Oils below 100 deg. C.	1.7
Oils 100-205 deg. C.	5.4
Oils 205-270 deg. C.	47.3
Soft pitch	35.9
Total	98.9

It contains nearly 50 per cent of distillable phenols, the fraction from 205 to 270 deg. C. being composed of phenols to the extent of 80 per cent. On mixing with formaldehyde and an alkali, this fraction polymerizes to a plastic substance even in the cold and on heating it yields a solid body. The tar is an interesting material, but its value is as yet entirely unproven.



## ALCOHOL IN STEAM FROM SODA DIGESTERS

A small pipe with a condenser was connected to the relief line from one of the digesters, and a portion of the steam allowed to blow off during the cook was condensed and tested for alcohol and acetone, with the following results:

## Composition of Condensate from Soda Digester

A—Using Hard Wood  
 Steam on at 11:30 A.M.  
 Up to 115 lb. pressure and relief valve opened at 1:15 P.M.

Hourly Sample	PER CENT BY VOLUME		Per Cent by Weight Ammonia (NH <sub>3</sub> )
	Methyl Alcohol	Acetone	
1	1.00	0.02	.....
2	1.48	0.06	.....
3	1.43	0.07	.....
4	1.37	0.07	.....
5	1.35	0.09	.....
Average	1.32	0.06	0.06

B—Using Bass Wood.  
 Steam on at 6:50 A.M.  
 Relief valve opened at 8:40 A.M.

Hourly Sample	PER CENT BY VOLUME		Per Cent by Weight Ammonia (NH <sub>3</sub> )
	Methyl Alcohol	Acetone	
1	0.62	0.03	0.026
2	0.80	0.04	0.026
3	0.44	0.04	0.023
4	0.59	0.04	0.021
Average	0.61	0.04	0.024

It was not feasible to measure the steam given off from each digester, but it was estimated at 145 gal. per digester. This would indicate a loss of about 2 gal. of alcohol and acetone from each digester of hard wood and about half that from each digester of bass wood. It did not seem feasible to condense any of the steam from the charge as it was dumped, but that given off immediately after the digester was discharged would undoubtedly have contained as much alcohol as that from the last sample during the cook. The amount of ammonia formed (0.06 per cent) is small but interesting. The values indicated here are too small to warrant recovery in an independent plant, but would probably be worth recovering as a side issue in a plant equipped for destructive distillation of the black liquors.

## COST OF OPERATION AND RETURN

The process discussed here merely replaces the rotary calciners by suitable retorts operating at temperatures well below a red heat. The multiple effect evaporators before the process and the leaches after the process need not be changed. The question concerns the cost of installation and operation of the retorts, condensers, etc., as compared with the rotary calciners. No fuel need be purchased to heat the retort since the carbon from the black ash will provide the necessary heat. One thousand gallons of 40 deg. black liquor contain only 3640 lb. of water and yield 2100 lb. of carbon after leaching which is in the ratio of 1.74 lb. of water per pound of carbon. The destructive distillation evolves heat so that the only work which the fuel has to perform is to heat the material to 600 deg. Fahr. and to vaporize the water. The



temperature of the stack gases is as low as that of the average steam boiler and on a basis of steam boiler performance a half of the carbon should be sufficient to heat the retorts and supply sufficient waste heat to dry the leached carbon in waste heat driers. It is therefore conservative to assume that if the carbon is not more valuable for other purposes it will be sufficient to cover all fuel requirements of the plant.

The absence of acid from the distillate makes it unnecessary to use copper in the condensers or stills and also eliminates the necessity for the complete distillation with high pressure steam or in vacuum to separate the acetic acid from the tar. The steam requirements

TABLE III—ESTIMATED COST OF OPERATION AND RETURNS FROM BY-PRODUCT PLANT BASED ON ONE CORD OF WOOD COOKED IN SODA DIGESTER

To be Credited to By-Product Plant per cord of wood:		
6.9 gallons 95 per cent methyl alcohol and acetone @ \$4.00	\$2.76	
44.5 lb soda ash (5 per cent of total, now lost in stack gases)	0.44	
570.0 lb carbon counted here only as fuel		
Saved by elimination of present rotary kilns	0.67	\$3.87
To be Charged against the By-Product Plant per cord of wood:		
Cost of plant estimated at \$1,170 for each cord per day—		
Labor	\$0.80	
Steam and power	0.35	
Superintendence, supplies and taxes	0.20	
Repairs at 10 per cent of cost of plant	0.33	
Depreciation at 10 per cent of cost of plant	0.33	2.01
Balance in favor of By-Product Plant per cord of wood		\$1.86

are therefore much lower than in a wood distillation plant. The chief difficulty remaining in the commercial development of the process is that of developing proper mechanical devices for opening and closing the retorts and for pushing the contents. Since a plant must be composed of large numbers of small units one man must be able to operate a machine handling a number of retorts simultaneously.

The estimated cost of operation and the returns from a by-product plant of this type are tabulated in Table III. The methyl alcohol and acetone figures are taken from the analytical data as 100 per cent strength and in the absence of actual data on refining losses, 5 per cent loss has been assumed and a further allowance made by not giving to the acetone a higher price than the methyl alcohol. The prices used in the table are those prevailing before the war. The carbon remaining after leaching the black ash is assumed merely to serve as fuel for the plant, though it has been shown to be saleable as a paint pigment and has interested the manufacturers of black gunpowder. No value at all has been assigned to the gases, or the tar which would serve as fuel if it could not be more advantageously utilized. The estimated saving of 5 per cent of the total soda ash in circulation is believed to be conservative, as is also the saving of labor, fuel, repairs, and depreciation on the present type of rotary kiln plant for calcining the black liquor.

#### Summary

It has been shown both by laboratory tests and by larger mill tests that the concentrated black liquors from the soda pulp process may be subjected to destructive distillation so as to yield 6.9 gal. of alcohol and acetone per cord of wood placed in the digester. The yield from a soft wood such as bass is as good as that from a hard wood such as beech. The only variation from the present process consists in replacing the rotary calciners by retorts in which the liquor is distilled at a temperature of about 600 deg. Fahr. If a 40 deg. Baume liquor is used the distillate contains about 4.4 per cent by volume of alcohol and acetone, no acid, and about 11.2 per cent by volume of tar which contains nearly 50 per cent of phenols. The black ash leaches readily to a clear liquid and the recovered carbon is sufficient to provide the necessary fuel for the whole by-product plant. The by-product plant will effect an additional economy by pre-

venting the present loss of soda which is carried as dust up the stack from the rotary calciners. The net saving by the introduction of a by-product plant is estimated, as shown in Table III, to be \$1.86 per cord of wood cooked in the digester.

University of Michigan.

### Current News and Notes

**Second Annual Convention of Technical Association of Pulp and Paper Industry.**—The second annual convention of the Technical Association of the Pulp and Paper Industry was held at the Waldorf-Astoria, New York, Feb. 6, 7 and 8, 1917, in conjunction with the general convention of the American Paper and Pulp Association. Tuesday, Feb. 6, was given over to executive committee meetings. On Wednesday morning a general session was held, and on Wednesday afternoon a session for the presentation of papers was held. A paper presented at this session by Profs. Alfred H. White and John D. Rue of the University of Michigan on methyl alcohol from spent liquors will be found printed in full elsewhere in this issue. On Thursday morning another session was held for the presentation of papers. The attendance was very good, and a lively interest was shown in all the subjects discussed.

**National Lime Manufacturers Meet in New York.**—The National Lime Manufacturers' Association held its fifteenth annual meeting at the Hotel Astor, New York, on Feb. 6 and 7. The president of the association, Mr. Wm. E. Carson, presided at the meetings. An interesting series of papers were read, taking up such subjects as revision of standard specifications for hydrated lime, discussion of barrels and packages, welfare work, trade acceptances, use of pulverized coal in lime kilns, group insurance, calcining of lime, continuous hydration and other subjects. G. F. Loughlin of the Geological Survey, author of the annual report on lime, gave an interesting talk on the work of the Survey in obtaining statistics. He said he was working up statistics on magnesium lime and dolomite, in view of their being the future raw materials for our magnesium industry. The members had a rare treat on Wednesday morning when Robt. Lee Montague of Montague, Va. ("42 miles from any railroad"), related a series of anecdotes of Virginia life. He is a natural talker, and his stories from life were rich in humor, and kept the audience in a continual uproar. On Wednesday morning a silver loving cup was presented to Lawrence Hitchcock for his work in establishing and successfully conducting the hydrated lime bureau.

**French National Laboratory of Physics and Mechanics.**—The Academie des Sciences, according to *Genie Civil*, has resolved to establish a National Physical and Mechanical Laboratory, for the purpose of scientific research directed toward industrial uses. The laboratory will be controlled by a council, of which half the members will be nominated by the Academy, one-fourth by the State Department, and the remainder by the chief industrial associations. The executive control will be in the hands of a small technical committee. Existing laboratories engaged on similar work will be affiliated to the National Laboratory, and will work in close relationship with the latter. Substantial funds will have to be provided for the working expenses of the laboratory, and for the assistance of the affiliated institutions.

**Dyestuffs Appropriation.**—The Senate has passed the Agricultural bill, which contains a provision for the appropriation of \$49,400 for research in dyestuffs. The money will be used by the Bureau of Chemistry.

## Air Drying

By Carl Hering

In some industrial processes the moisture in the air is objectionable, either because the water is an undesirable foreign material, or because it interferes with the use of the air for drying operations; its variability is another objection, as also its tendency to promote bacteriological growth; extreme desiccation acts as a food preservative. In the operation of drying objects in the air, the time required increases rapidly as the air is more nearly saturated, and the objects to be dried are, of course, never any drier than the air which was last in contact with them and which is, therefore, approaching its saturation point on account of the added moisture from the objects. Moreover, the evaporation of the moisture in the objects produces a lowering of the temperature, and this in turn lowers the drying power of the air because the lower the temperature of air carrying a definite amount of moisture, the more nearly is it saturated. Replacing the air rapidly or heating it, are the usual methods for diminishing these objections, but in some cases, as for instance, in the drying of grain, foodstuffs, photographic films, etc., the high temperature and the dust carried by moving air are both objectionable, and on warm, humid days in summer it is sometimes physically impossible to dry such objects properly.

In blast furnaces the water carried by the air robs the furnace of a certain amount of heat, as it must be heated to those high temperatures and is no doubt dissociated also. On a humid summer day, for instance, the amount of water entering a modern blast furnace with the air is about 4 to 5 gallons per minute, which is about equal to what will run out of a large house spigot. It is, however, not quite as bad as though such a formidable stream of water were forced as water into the furnace, as is sometimes alleged, because in the form of vapor in the air the water already contains its latent heat of vaporization, and therefore does not rob the furnace of that formidable amount of energy. In blast furnaces one of the chief objections to the moisture is its variability, making it difficult, if at all possible, to get uniformity of the iron produced.

In the fires of steam boilers this moisture robs the fire of the heat which this water vapor carries off as a dead gas into the chimney. Those who claim that by being dissociated into oxygen and hydrogen at those high temperatures, it adds valuable combustible materials, seem to forget that it first had to rob the fire of exactly as much energy for its decomposition as these two gases give up on uniting again, as they seem to appear in the chimney as steam.

Damp air being very bad for consumptives and as it encourages bacterial propagation in general, it might sometimes be desirable to dry the air in rooms for sanitary purposes.

There are, therefore, cases in which it is of great importance to desiccate the air to a greater or less extent, and many others in which it would be desirable if the cost is not prohibitive. It is, therefore, often a question of the calculation of the cost, and as this cost increases with the degree of extraction of the water, it is also a matter of calculation to find how far it pays to go in the degree of drying.

A number of methods are available. Heating the air reduces its degree or percentage of saturation, and it, therefore, takes up more water with greater facility; and if heated under constant pressure, as for instance, under atmospheric pressure, it reduces the amount of water per cubic foot of the air because the air has expanded; but it does not diminish in the least the

amount of water in a given quantity of air, both measured in pounds, and is therefore, not a process of drying the air.

Reducing the pressure by creating a partial vacuum, increases the drying capacity of the air because the water in the objects then tends to vaporize more readily. The low pressure air itself is really wetter then, in that it contains more water per cubic foot or per pound; hence this is only a process of drying objects, but not of drying the air itself. The water may be said to be sucked out of the objects.

Compressing the air deposits some of the vapor in the form of water and is therefore a true partial desiccation. Such compression, however, also heats the air which itself tends to lower the degree of saturation, and the latent heat of the water which has been condensed, must also appear somewhere as sensible heat. If cooled while compressed in order to get out more of its water, the air will be still colder when expanded again, and this increases its degree of saturation until reheated. Moreover, at the low temperatures produced by expansion there is great danger of ice forming in the valves, which clogs them. This process, therefore, involves complications besides the transferences of heat and the use of relatively large moving machinery; its application is, therefore, limited.

Liquefying the air and then evaporating it again dries it absolutely, as the moisture (besides the oxides of carbon) is all frozen in the process and can be filtered out as a solid. It is one of the few methods for producing absolute dryness, but is expensive, though by using a suitable heat regenerator this cost ought to be very greatly reduced and perhaps it might then be commercial; absolute dryness, however, is seldom required.

Concentrated sulphuric acid, solid chloride of lime, and other moisture absorbing materials, are simple and effective ways of very slowly drying a small amount of confined air, and are much used on a small scale, to which, however, the cost limits them. They are said to absorb even extremely small quantities of moisture and may, therefore, be classed among the absolute methods.

Centrifuging the air at very high speeds ought to concentrate the moisture in one part, and perhaps to oversaturation, thereby rendering the other part drier; but the expense would probably be prohibitive.

But of all the methods of drying, the one based on cooling is the most practical one when dry air is required on a relatively large scale. When the temperature of air containing moisture is reduced to below the so-called dew point (the temperature at which that amount of moisture saturates it) the excess of moisture above saturation at that lower temperature, is condensed as water or ice and may, therefore, readily be separated. At that lower temperature it is then saturated, which means that it will hold or take up no more water, but if heated again to its original temperature after the excess of water has been separated from it, the air will be drier to the extent of the amount of water or ice which has thus been condensed from it; and the lower this temporary temperature was, the greater will have been the extraction, though not in proportion. While this is only a partial desiccation, unless carried to an extreme like in liquid air, yet with modern ammonia ice machines it is theoretically possible to so reduce it that when heated again to normal temperatures, the humidity will be only 2 to 4 per cent, which might well be termed "bone-dry air" and which probably never exists naturally.

As stated above, the applicability of this method generally depends on the cost, and this cost varies with the degree of extraction; hence a predetermination of this cost becomes important. Except in the simplest



cases the calculations are quite involved when all the factors are taken into consideration, and when precision is desired; they should, therefore, not be attempted by those who do not understand them. But roughly approximate calculations are easily made with the aid of conveniently arranged tables.

The principles involved in this process, on which the calculations are based, are as follows: Atmospheric air always contains some water, though it is rarely saturated. The amount of water, termed the humidity, is usually stated in per cent, which is 100 times the ratio of the actual amount to the amount it would contain at that same temperature if saturated; it is measured with the wet and dry bulb thermometers, the readings of which are interpreted by means of tables, as is well known. Care must be taken with the measurements made with the wet bulb, as the rate of fanning or "ventilating" has an important effect if insufficient.

To dry this air it must be cooled first down to the so-called dew point, that is, the temperature at which the water which it contains saturates it. In this first stage of the process the moist warm air follows the laws of gases, and the energy required to be extracted is determined from the specific heat of air and that of the constant amount of moisture contained.

If then cooled below this saturation temperature it begins to deposit its water, and from there on down the air is always saturated at the respective temperatures, hence it no longer follows the laws of gases, but those of saturated vapors. During this stage the energy to be extracted consists of three parts, the two parts necessary to cool the air and the water in it, and the third, the latent heat of as much of the water as has been condensed. If this temperature is below freezing there must be added a fourth part, the latent heat of the amount of ice formed.

Of these parts by far the two largest ones are those for the cooling of air and for the condensation, and of these the former is generally larger than the latter. The freezing energy is relatively much smaller and that to cool the moisture is a small amount, only a few per cent.

After the extraction the air with whatever moisture it still contained at saturation at the lowest temperature must then be reheated which requires a formidable amount of heat. This might be taken from the atmosphere through thin walled metallic ducts and air circulating devices; though the calories then cost nothing, it may be cheaper to heat it more quickly with a heater.

When this dry warm air is then used merely as combustion air in furnaces, the above are all the chief factors entering into the calculations. The duty imposed on the refrigeration machine then is to carry off those calories which must be extracted and which might be termed negative calories, while the duty on the heater is to provide the reheating of the dry air.

But when the dry air is used for drying wet objects, like photographic films or grain, there is an additional and sometimes quite important factor. The vaporization of the water in the objects consumes latent heat. If the drying is done in closed chambers, as it naturally would be, this latent heat will be extracted chiefly from the air and the objects, and will, therefore, cool both. This in turn reduces the drying power of the air and, therefore, the speed of drying. It would also be conceivable that the cold produced by this latent heat energy might freeze the objects; at a uniform temperature of 32° F. the evaporation of each pound of water would freeze another pound. Hence, in such cases the dry air must either be first heated to such a temperature that it will supply this latent heat without cooling itself too much, and this might involve heating it to

a rather high temperature, or if the objects would be injured by this temperature, the air must be heated during the drying operation, or the drying be slow enough to give time to absorb heat from the surroundings.

The cost of this process of drying the air being generally the criterion to decide whether and to what degree it may be practicable to use it, and as this cost may be prohibitive, thermal economies in the process may become quite important, particularly in saving the duty of the refrigerating machine, negative calories, at least at cold temperatures, being far more costly than positive ones in a heater. An accumulation of economies in different parts of the process may together amount to an important saving. An analysis of the most perfect conditions may, therefore, be useful as a guide.

The formidable amount of energy, generally more than half of the total, which must be extracted from the air itself, aside from the water, is, of course, the same in amount as that necessary to reheat it again afterwards, differing only in its sign. It is, therefore, theoretically recoverable by means of a regenerator. If the original warm, moist air be made to pass in one direction through channels with thin metallic walls, and the cold dry air be made to pass in the opposite direction on the other side of those metallic walls, this energy will be exchanged through the thin walls, whereby the ice machine is relieved of that much of its duty, which may amount to about a half; as the moist air then arrives at the refrigerator at a cold temperature, the ice machine would have to supply only the difference; the reheating of the dry air is then also largely saved, as it would come out at nearly the temperature of the incoming air.

Moreover under suitable circumstances much of the water, probably about half or over, would then come out in this regenerator as water, thereby considerably reducing the amount of ice formed and saving its latent heat. As the object is to separate the water from the air, it is taxing the refrigerator unnecessarily to further cool this water after the separation has been effected, and as this usually begins well above the freezing point, some further energy could be saved by having it run off as soon as formed, that is, at the temperature of formation. This would take place in such a regenerator when appropriately constructed.

The cold dried air from the refrigerator cannot, however, take up any more heat in this regenerator than will heat it to the temperature of the incoming air, while the warm moist air could convey to it not only its own heat, but also all the latent heat of the water which is condensed in the regenerator. Hence, additional economy could be obtained, if desired, by cooling the air somewhat more while passing through the regenerator by means of the partially spent cooling liquid or gases coming from the refrigerator. This would take out more of the moisture as water, instead of as ice, and the colder calories of an ice machine cost more than the less cold ones.

In an ammonia ice machine very low temperatures necessarily exist, and as the air will be drier the colder it has been, it would be desirable to make use of these very low temperatures or at least to approach them. This becomes practicable when such a regenerator is used, as the ice machine can then absorb nearly all its calories at quite low temperatures. Makers of ice machines say that -15° F. (-26° C.) can be obtained in regular operation. Cooled to this temperature and then heated again to 70° the air will have a humidity of only 2.95 per cent, hence is extremely dry air. Liquid ammonia boils at -28.6° F. (-3.7° C.), hence as -15°



leaves a margin of 13.6° F., still drier air might be obtained, though the difficulties and the thermal losses increase as the temperature is lower.

Makers say that the efficiency of an ammonia ice machine is reduced 25 per cent by using brine as the conveyor of the cold. Using the ammonia directly would, therefore, increase the capacity of an ice machine 33 per cent and by using refrigerating coils made without joints by autogenous welding, the danger of leakage in the refrigerating chamber is avoided, and this would make the direct use of the ammonia practicable. It has the objection that steam cannot then be passed through the coils to melt off the ice, as that would contaminate the ammonia with water. But these coils when loaded with ice could for a short time or alternately be used as the ammonia condensers, thereby melting the ice, and at the same time making good use of the negative calories in it.

A common way of producing moderately dry air is to spray it with very cold water. The advantage of this method is its simplicity, the disadvantages are the large quantities of water required to be cooled and forced through the spray nozzles, and that the air is not very dry, though uniformly so. The theoretical maximum dryness would then be 2.11 grains of water per cubic foot or about 0.3 lbs. per 1000 cubic feet at 32° F., and when this is then heated to say 70° F., the air would have a humidity of 24.5 per cent, containing about 0.28 lbs. per 1000 cubic feet, which is not very low, though probably low enough for some purposes.

But these figures represent theoretical perfection and it would require an infinite amount of cooling water. This cooling water must absorb considerable energy in reducing the temperature of the air and in condensing the water, and this energy will, of course, heat this water; moreover the coldest temperature of the air will be that of the cooling water when it is warmest, as it will be the temperature of the water last in contact with the air, that is after it has absorbed this energy. Water, as such, cannot be cooled lower than 32° F., and to cool the air down to say 38° F., would allow only 6° rise, hence enough cold water at 32° would have to be used that it will absorb all this energy and not rise more than 6° F.; the amount will be roughly inversely proportional to this rise. When thus cooled to 38° F. and then heated to 70°, the humidity of the air will be 31.1 per cent, containing about 0.355 lbs. per 1000 cubic feet, which is about 78 per cent of the perfect extraction referred to above.

Assuming the original moist air to be at 70° F., and to have a humidity of about 75 per cent, therefore containing about 0.855 pounds of water per 1000 cubic feet, which is not unusual on a moist day in summer, the cooling water would have to take up a little over 1000 B T U per 1000 cubic feet of air. And as the water is allowed to be heated only 6° F., this means 167 lbs. or about 20 gallons of cooling water per 1000 cubic feet of air for perfect operation. For a blast furnace requiring 45,000 cubic feet per minute it would, therefore, take 900 gallons of cold spray water per minute. The amount of water extracted would be 0.50 lb. per 1000 cubic feet, which is 58.5 per cent, or only a little over half, of what was in it, leaving 41.5 per cent of the water still in the air.

Some improvement in this method may be obtained by passing the air through successive sprays so that it might finally reach more nearly the lowest temperature, 32° F. And the spent spray water of the coldest spray may be used over again for the next warmer one, and so on. A lower temperature than 32° and still have unfrozen spraying water, can be obtained in this method by using a brine for the spray water; the extracted

water will then necessarily gradually dilute the brine which must, therefore, be concentrated again, as by boiling. Using the same water over again also means that it must be filtered, as it contains all the dirt of the air and this might otherwise clog the small spray nozzles. Whether such economies are worth their expense is a matter of computation.

An important advantage of this spraying method is that the air and the cooling water are brought into direct and intimate contact with each other, hence the transference of heat is rapid, and ought to be very nearly perfect. It is for this reason sometimes called the direct method. Whether this advantage is too dearly bought is a matter of computation, and depends also on the particular conditions of the case.

A simple and convenient method of carrying out this cooling process on a small scale is by forcing the warm moist air through cracked ice. If carried out to the extent that the air leaves at 32° F., it will then contain a trifle more than 0.3 pounds of water per 1000 cubic feet, and when then heated again to 70° F., it will have a humidity of 24.5 per cent and contain about 0.28 lbs. per 1000 cubic feet. All the water then leaves at 32° F. and assuming the ice to be no lower than 32°, the extracted heat will all be the latent heat of the ice melted, which is about 144 B T U per pound. Assuming the original air to be 70° F. and to have a humidity of 75 per cent, the cooling will, under perfect conditions, require the extraction of about 1280 B T U, hence the melting of 8.9 pounds of ice, per 1000 cubic feet. Some further economy in this method can be obtained, if desired, by passing the air first through granular material over which the melted ice and extracted water are made to flow or spraying this water into the air before it reaches the ice. Regeneration of the heat in the air itself will add further to the economy. As no useful effect results until the saturation temperature is reached, the ice is being consumed uneconomically until the temperature reached is low. While it costs less ice the lower the original humidity, it is by no means in the same proportion.

For rough preliminary estimates the accompanying Table I may be of interest. It has been reduced to

Table I.—1000 Cubic Feet of Saturated Air.

Temperature F.	Pounds of Water	B.T.U. to Reduce from Given Temperature to Water at 32°	B.T.U. Per Pound of Water Extracted	B.T.U. to Reduce from 32° to Given Temperature	B.T.U. Per Pound of Water Extracted
100	2.82	3,840	1,520	...	...
90	2.11	2,910	1,610	...	...
80	1.56	2,150	1,710	...	...
70	1.14	1,530	1,830	...	...
60	.821	1,030	1,980	...	...
50	.582	604	2,160	...	...
40	.407	256	2,440	...	...
32	.302	0	...	0	...
30	.276	...	...	29	...
20	.176	...	...	358	2,860
10	.111	...	...	614	3,210
0	.0687	...	...	843	3,620
- 10	.0407	...	...	1,060	4,060

1000 cubic feet of saturated air at the given temperatures. The second column gives the contents of water in pounds. The third gives approximately the thermal units required to be extracted in order to reduce the temperature from that given in the first column to 32° F. without freezing any of the water; hence it refers to the best that could be obtained by a single spraying with water at 32°, or with passage through cracked ice. It includes the cooling of the air with its water, and the latent heat of condensation. The figures are only approximate and do not include the smaller factors. The fourth column gives the corresponding rates per pound of water extracted in this way. The fifth

is like the third, except that the cooling begins at 32° and ends at the temperatures given in the first column; it therefore assumes that all the water that condenses at 32° has already been extracted as such; these figures must, therefore, be added to those in column 3 if any freezing is done, and it is here assumed that all the free water at 32° is drained off and is not to be frozen. The sixth column gives the corresponding rates of extraction. The amount of water left in the air can be obtained from the second column and will, therefore, be 0.302 lbs. for all the values in column 4, while for column 6 it will be that given in the second column for the temperature to which the reduction has been carried. As the figures are based on the physical laws and constants the inefficiencies in the process must be allowed for in addition.

If the water is all frozen, which is sometimes done, an additional number of B T U will be required to freeze that which would have run off as such at 32°. These figures assume saturation, hence the B T U required to first reduce the air to saturation must be added. If regeneration is used the B T U in columns 3 to 5 will be considerably reduced; a further reduction in B T U would be obtained by cooling in steps; the figures for the B T U are therefore not the lowest possible.

It will be noticed from column 2 that the amount of water extracted for every 10° drop is far greater at the higher than at the lower temperatures. Also that the cost in B T U for extracting a pound of water increases at the lower temperatures, though perhaps not as greatly as might have been supposed.

For saturated air at 60° F. the B T U to cool the air to 32° are roughly equal to those required to condense the water; above 60° those for the latter form the greater half, while below 60° this is reversed.

Makers of ice machines rate them in tons of ice per 24 hours, which they say means that the machines will do as much cooling as that much ice at 32° F. would do in melting to water at 32°, hence this corresponds to the latent heat of melting ice. The latter is about 144 B T U per pound, hence per ton of 2000 lbs. per 24 hours the rate of cooling produced by an ice machine would be an extraction of 200 B T U per minute; this is based on the use of brine as the conveyor; when the ammonia is used directly the capacity is said to be a third higher. From tests of large ice machines using brine, it would require a little more than one kilowatt to operate an ice machine generating 200 refrigerating B T U per minute.

Philadelphia, Pa.

**Magnesite Deposits Being Worked in Washington.**—The American Mineral Production Company has begun the production of magnesite and various magnesium salts from large deposits owned by the company in Stevens County, Washington. The deposit lies 5 to 10 miles west of Valley, a small town on the Great Northern, about 50 miles northwest of Spokane. It is exposed in several places to widths of 30 to 600 ft., and is estimated to contain an enormous quantity of magnesite, consisting of a coarse to finely crystalline material of reddish to pinkish color. It is not mixed with as much limestone or dolomite as the Australian magnesite. Analyses across wide areas are as follows:

	Silica	Alumina	Iron Oxide	Lime	Magnesium Carbonate
No. 1	0.80	0.40	2.70	3.30	92.10
No. 2	0.89	none	.58	none	95.00

A number of chemists and engineers have been working for some time on the property, and it is claimed by the company that it is pure and in larger quantities than the Austrian, which formerly produced 90 per cent of the world's production of this grade of magnesite.

## The Effect of Centrifugal Force on Colloidal Solutions\*

By Eugene E. Ayres, Jr.

Considerable work has been done on certain centrifugal separations which cannot be accomplished by the highest degree of centrifugal force at present capable of commercial application. Some chemists have expressed surprise that gasoline cannot be separated from kerosene, or that soap cannot be mechanically removed from aqueous solution. Others are inclined to doubt that colloidal gold can be successfully deposited from a clear gold sol.

There is one certain method of eliminating guesswork as to the practicability for a given set of conditions—and that is the experimental method. If a careful trial succeeds or fails, the practicability for that set of conditions is no longer a matter of opinion. But pertinent questions may still be asked. For instance, will the result secured by the test represent the best that any type of centrifugal machine may be expected to secure? Is it possible that a higher degree of force can actually remove anything unaffected by the lower degrees? Is it possible that, although complete separation may have been secured, the components are not separately discharged? Such questions can be correctly answered only by thoroughly understanding the complexity of the task confronting the centrifugal separator.

**The Colloid State.**—Let us first review briefly a few general aspects of colloid chemistry.

The term *dispersoid* has been correctly applied to systems involving every size of particle, from the macroheterogeneous suspension to the true molecular or ionic solution. The term *colloid* in its exact sense designates a state of matter with respect to degree of dispersity within the rather narrow limits defined by sub-micro- and amicro-heterogeneity. The largest colloid particles in this sense are about 0.1 $\mu$  in diameter—at the extreme limit of microscopic visibility, and at about the limit of retention by the best clay filters.<sup>1</sup> Particles smaller than 0.1 $\mu$  may be seen by use of the ultramicroscope, whose lower limit is about 3 $\mu$ . Colloidal phenomena are exhibited by dispersoids whose particles are anywhere from 0.1 $\mu$  to 1 $\mu$ . Most solutions, or molecular dispersoids, are just beyond this limit, a fair average molecular diameter being about 0.1 $\mu$ , although some of the more complex molecules are much larger.<sup>2</sup>

All these systems are polyphasic, but the disperse phase will confer various sets of properties upon the dispersoid, depending upon the specific surface. Therefore the complex system involving many different degrees of dispersion may be said to have distinct phases corresponding to the three series:

1. Particles larger than 0.1 $\mu$ .
2. Particles between 0.1 $\mu$  and 1 $\mu$ .
3. Molecules.

The second division defines the colloid state in its strictest sense. But many of the phenomena that characterize the true colloid characterize to a greater or less degree the coarser dispersoids, whose particles may be even larger than 5 $\mu$ , and can be retained by the ordinary filter paper.<sup>3</sup>

\*A paper read at the Ninth Annual Meeting of the American Institute of Chemical Engineers, New York City, January 12, 1917.

<sup>1</sup>The  $\mu$ , or micron, is 0.0001 cm.

<sup>2</sup>The  $\mu$  is 0.001 $\mu$  or 0.000001 cm.

<sup>3</sup>Wo. Ostwald-Fischer, "Handbook of Colloid Chemistry," p. 263.

<sup>4</sup>The diameter of the starch molecule is said to be about 5 $\mu$ .

Lobry de Bruyn and Wolff, Rec. Trav. Chim. Pays-Bas., 23, 156 (1904).

<sup>5</sup>Ordinary filter paper holds back particles of about 5 $\mu$ ; a hardened filter (Schleicher and Schull, No. 602 e. h.), those about 2 $\mu$  in diameter. (Wo. Ostwald, l. c.).



Certain classifications of dispersoids have been made without regard to degree of dispersion. It will be unnecessary to discuss these distinctions here except to mention the colloids that tend to form a gel. There are several characteristics of these gel-forming dispersoids, especially affecting centrifugal separation. The particles are usually extremely small—even beyond the limit of the ultramicroscope—and the disperse phase and the dispersion medium are composed of both components.<sup>5</sup> The solid gel has an enormous viscosity fac-

Temp.	Concentration Agar.	
	Liquid.	Solid.
36°	0.47	3.2
5°	0.99	

tor. This would prevent any centrifugal separation even under the most favorable conditions. When the gel is warmed, to liquefy and reduce viscosity, the size of the particle is even further reduced, and the compositions of the two phases tend more nearly to equalize in percentage of components. When this equilibrium is approximately established, the absolute density of the disperse phase is therefore nearly zero.

We shall put aside the gel-forming colloid for the simpler case of the suspensoid whose particles, whether rigid or deformable, have little chemical or solvent affinity for the dispersion medium.

At first glance, it might appear necessary to limit our problem still further. In the case of separation by gravity, the degree of dispersion is likely to change enormously on account of some of the influences commonly known to affect stability. Since time, in the case of centrifugal separation, is a matter of minutes rather than days, we can eliminate these factors.

**A Few References.**—With reference to the theme of this paper—the study of colloids under the influence of centrifugal force—the first task will be to formulate a mathematical expression of the phenomenon of subsidence. The subject has already been treated in a variety of ways.

The probable influence of gravity on solutions was studied by Gay Lussac. He examined the upper and lower portions of a salt solution in a tube two meters long maintained at a constant temperature for a long time. The results were negative. In 1887, Gouy and de Chaperon, commenting on this work, demonstrated theoretically that the force of gravity was too small. L. Vegard<sup>6</sup> from thermodynamical considerations, gives an expression for the equilibrium of a solute. H. S. Davis<sup>7</sup> gives a mathematical analysis of the effect of gravity on a solute, by applying Fourier's law of linear diffusion. His experimental results are also negative.

One of the earliest of the difficult centrifugal separations was made as long ago as 1895 on a mixture of two gases—hydrogen and hydriodic acid.<sup>8</sup> The gases were whirled for three hours in a machine operated at 2400 r.p.m. with a radius of rotation of 21 centimeters. A 3 per cent difference in concentration was noted.

About twenty years ago, it was observed that subjecting a salt solution to moderate centrifugal force will cause a measurable difference of potential between the center and the periphery. More recently<sup>9</sup> a centrifuge was successfully employed to obtain a concentration of antitoxic substances in immunitive serums. Perhaps the most striking work on the centrifugation of solutions was done by Van Calcar and Lobry de Bruyn<sup>10</sup> who were able to crystallize out about three-

eighths of the salt from a saturated Glauber's salt solution.

**The Problem.**—When any dispersoid is subjected to centrifugal force—no matter in what type of apparatus—the separation of any individual particle will depend upon the distance through which it must move in a given time before being considered "separated." There are two types of centrifugal machines. In one case, a container is filled with liquid and rotated. The particle must move until it is held in firm contact with the bottom of the vessel before any separation can be measured. In the other case, the liquid is passed continuously through a rotating cylinder and it is possible to draw the liquid from the point nearest the center of rotation while the machine is still in motion. Separation can therefore be detected when the particle has been moved only a fraction of the distance from the surface to the bottom. Our consideration will be limited to this more convenient type.

It would seem that the whole problem hinges upon the proper calculation of the velocity of a suspended particle acted upon by a force. But what is the force? When we think of velocity, we naturally turn to the law of Stokes.

$$U = \frac{f}{6\pi r\eta}$$

where  $U$  is velocity of the particle of radius,  $r$ , acted upon by the force  $f$ , while the viscosity of the medium is  $\eta$ . The formula is based upon the conception that a small body, acted upon by a constant force, will move through a resisting medium with a constant velocity. The normal acceleration is neutralized by friction. The law was applied first to the fall of raindrops through the air. More recently, a large number of important investigations have been based on Stokes' equation, and its accuracy has been verified for liquid as well as gaseous media, and for colloidal particles as well as raindrops.

J. Perrin<sup>11</sup> showed the law to be valid for particles as small as one-tenth of a micron. Einstein,<sup>12</sup> in his work on the coefficient of diffusion, assumed Stokes' law to be correct for molecules larger than one-thousandth of a micron, and his results show his assumption to be correct. On the other hand, M. Pellat,<sup>13</sup> in his investigation of ionic translation in electrolysis, gives some indication that Stokes' law will require a slight modification when applied to particles as small as the potassium ion. Of course our chief interest is in the relatively large particles to which the law has been found to apply without correction.

We are told, then, that colloids will settle by gravity according to a given formula. Emil Hatschek<sup>14</sup> offers a numerical example in the case of a gold sol, and utilizes the very evidently incorrect result as an interesting clue to certain other factors that make for the stability of colloids.

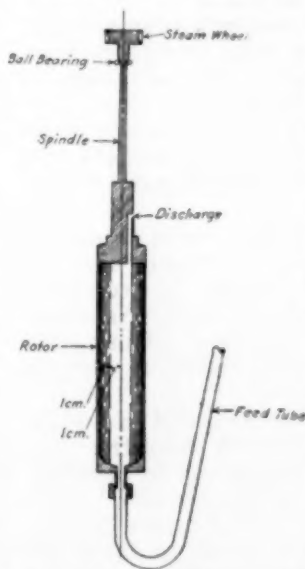


FIG. 1—CONTINUOUS CENTRIFUGE

<sup>5</sup>Hardy, Proc. Roy. Soc., 1900, 66, 96. Agar gel (2.23 per cent).

<sup>6</sup>Contributions to the Theory of Solutions. (Phil. Mag., Series 4, No. 77, p. 258.)

<sup>7</sup>Transactions of the Nova Scotia Inst. of Science. Vol. XII, Part 3, pp. 291-301, 1912.

<sup>8</sup>Bredig, Zeitsch. f. physik. Chem., 17, 459.

<sup>9</sup>R. P. Van Calcar, Voir. Verslagen, March 19, 1904, p. 842.

<sup>10</sup>Rec. Trav. Chim. Pays-Bas., Vol. 23, pp. 218-223, 1904.

<sup>11</sup>"Brownian Movement and Molecular Reality."

<sup>12</sup>Ann. der Physik., 1906, XIX, 289.

<sup>13</sup>Traité d'Electricité, Vol. III, p. 56.

<sup>14</sup>"Introduction to the Physics and Chemistry of Colloids," p. 24.



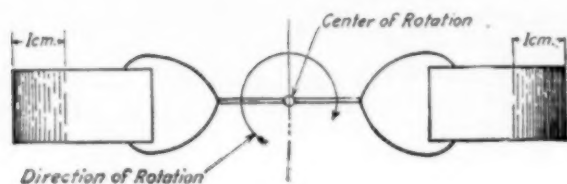


FIG. 2

What are these other factors? It is true that if precautions are taken, as recommended by Perrin, Wo. Ostwald, and others, to avoid small convection currents by observing the rate of fall in a capillary tube, the initial rate of fall will correspond to the equation. But after a time the rate will retard and then cease altogether. In other words, the force of gravitation has been neutralized by other forces acting upward, and the system is at equilibrium.

**The Dynamics of Colloids.**—Just as Van 't Hoff has been the pioneer in developing the analogy of dilute molecular dispersoids to gases, so M. Jean Perrin has done the first work in extending the kinetic theory to colloidal and even subcolloidal degrees of dispersion. Van 't Hoff had shown that the molecules of a solute in dilute solution play the same part as the molecules of a gas—develop the same kinetic energy, and exert a similar pressure on the walls of a semi-permeable membrane.

The earlier work on the osmotic pressure of colloids indicated a small but invariable value—sometimes so small as to raise a suspicion of experimental error or impure sols, but so invariable as to lead finally to the conclusion that all colloids did exert a definite osmotic pressure. Perrin showed that the osmotic pressure of colloids is due to their own kinetic energy, an energy derived from the molecular energy of the dispersion medium by molecular impacts on the colloidal particle. Kinetic energy implies motion, and colloids are found to possess an irregular, transitory motion known as the Brownian movement, from the name of its discoverer.

Osmotic pressure is thus a property common to all dispersoids whose particles are small enough to show Brownian movement. The largest particles showing this movement are about  $10\mu$  in diameter—just about visible to the unassisted eye—but their osmotic pressure is negligibly small. In fact, some particles will settle completely by gravity when less than one micron in diameter.

A second property of some dispersoids is an electric charge. The charge may be either positive or negative, and may be caused either by absorption of electrolytes or by an actual ionization of the particles. Naturally, the presence of equal electric charges of similar sense would seem to lead to an electro-repulsion between particles.

Osmotic pressure can be easily measured in a given case by the usual methods of physical chemistry. The size and sense of the electric charge can be measured by the speed of migration in an electric field.<sup>10</sup> This has been done and the conclusion was drawn that the electro-repulsion cannot be great enough to affect subsidence, although it greatly influences the rate of what is termed "agglomeration crystallization." So there remains only the osmotic pressure.

Instead of measuring this pressure and in some way computing its upward component, it is more convenient to start with the law of distribution derived for colloids by J. Perrin.

**The Law of Distribution.**—Imagine a liquid containing a number of small particles of uniform size and distributed uniformly throughout the liquid. After the system has been allowed to stand for a time, the particles will have arranged themselves in a definite order. The concentration (number of particles per unit volume) will be greatest at the bottom, and will vary in an exponential manner with the height according to the equation:

$$\log_e \frac{c_0}{c_x} = mgkx,$$

where  $c_0$  is the concentration of particles at the initial level,  $o$ , (see Fig. 3),  $c_x$  is the concentration at the height  $x$ ,  $m$  is the mass of the particle,  $g$  is the acceleration of gravity,  $k$  is a constant.

If we put

$$mgk = \alpha,$$

we may write the expression

$$c_x = c_0 e^{-\alpha x},$$

In order to determine the concentration at any point,  $x$ , we assume an empirical initial concentration—in

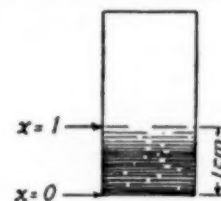


FIG. 3

other words, we take a value,  $k$ , to represent the number of particles in a unit volume when the system was uniform.

we can now write

$$k = \int c_x dx = \frac{c_0}{-\alpha} e^{-\alpha} + c'.$$

The depth of liquid is conveniently taken as 1 centimeter. We can therefore integrate between the limits, 1 and 0.

$$k = \int_0^1 c_x dx = \frac{c_0}{-\alpha} [e^{-\alpha} - 1],$$

whence

$$c_0 = k \left( \frac{\alpha}{1 - e^{-\alpha}} \right),$$

and

$$c_x = k \left( \frac{\alpha}{e^{\alpha x} - e^{\alpha(x-1)}} \right).$$

By the substitution of any value of  $x$  between 1 and 0 we can find the concentration at  $x$  as a function of  $k$ , and  $\alpha$ .

You will remember that

$$\alpha = mgk.$$

$k$  is a constant equal to Avogadro's constant divided by the product of the perfect gas constant and the absolute temperature. Assuming a normal temperature, the constant  $k$  is about equal to  $3 \times 10^{18}$ , in c.g.s. units. The mass,  $m$ , is equal to the volume times the absolute density,  $d$ .

$$m = \frac{4}{3} \pi r^3 d;$$

$$\alpha = \frac{4}{3} \pi r^3 d \cdot 980 \cdot 3 \cdot 10^{18};$$

$$\alpha = 10^{18} \cdot r^3 d.$$

For particles of different sizes and densities,  $\alpha$  can be easily computed and substituted in the formula for concentration, from which the condition at equilibrium

<sup>10</sup>The Svedberg, Koll.-Zeitschr., Vol. VI, 238 (1910).

can be shown for any value of  $\alpha$ . It is more convenient to express  $k_1$  as a weight percentage.

The following tabulation will give a fair idea of the various arrangements with different values of  $\alpha$ .

$\alpha$	$c_1$	$c_2$	$c_0$
1	0.58	1.04	1.60
10	0.001	0.10	10.00
100	10 <sup>-48</sup>	10 <sup>-20</sup>	100.00

In each case, the initial concentration,  $k_1$ , was taken as 1 per cent.

Similar values can be easily computed for any other values of  $\alpha$  or for smaller or larger values of  $\alpha$ , or for other values of  $k_1$ .

It is seen that the arrangement at equilibrium is a function of  $\alpha$ . But  $\alpha$  is the product

$$10^{\alpha} r^2 d$$

so that the radius of the particle that will give a certain  $\alpha$  will depend upon the absolute density,  $d$ . Thus, when  $\alpha = 1$ , a particle of low density must have a radius of 10<sup>-6</sup> centimeter, whereas if the density is high, the radius must be 10<sup>-6</sup>.

The condition represented by  $\alpha = 100$  leaves no room for doubt. Gravity separation will be complete. In such a mathematical application, the liquid can never have a concentration at any point equal to absolute zero, because in such a case the equation for distribution would be indeterminate. But such concentrations as, for instance, 10<sup>-20</sup> per cent, can be considered as practical zero. The condition represented by  $\alpha = 10$ , also means a very thorough separation. When  $\alpha = 1$ , the separation, though not at all complete, is still easily measurable.

It is interesting to note that Zsigmondy found the gravity subsidence of a gold sol to be measurable when the particles were about 75 $\mu$  in diameter. Such a particle with the high density of gold would make  $\alpha$  equal to about 1. Occasionally we come across statements that particles usually will not settle by gravity when as small as 0.1 $\mu$ . Theoretically the condition of equilibrium shown for  $\alpha = 1$  should obtain unless the electromotive forces should bring about agglomeration crystallization. In this case, subsidence will be complete.

We might stop to compute the effect of gravity on the distribution of true solutions where  $\alpha$  is very small, but we must go on to apply the formulæ to centrifugal machines.

If it were possible to construct a centrifuge with an infinitely long radius of rotation, we could use precisely the same formulæ for centrifugal separation as for gravity subsidence. But, given a constant r.p.m., the force varies directly with the radius of rotation, and the force is greater at the periphery, 0, than at the surface, 1. In Fig. 4, let the distance from the center of rotation to the periphery be designated by  $R$ . The radius of rotation of any point,  $x$ , may be expressed by  $R - x$ . Therefore, we have

$$\frac{f_0}{f_x} = \frac{R}{R-x},$$

$$f_x = f_0 \frac{R-x}{R},$$

where  $f$  is the factor multiplying the acceleration of gravity. The centrifugal force,  $f_x g$ , at any point,  $x$ , is equal to a function of  $x$  involving the centrifugal force,  $f_0 g$ , at the periphery.

The general expression for distribution in the case of centrifugal force is

$$\log_e \frac{c_0}{c_x} = m f_x g k x,$$

or

$$= m k f_0 g x \left( \frac{R-x}{R} \right).$$

$$\text{Let } \beta = m k f_0 g,$$

$$\text{and let } k_1 = \int c_x dx = c_0 \int e^{-\beta x \left(1 - \frac{x}{R}\right)} dx.$$

This function cannot be actually integrated by the ordinary calculus. But approximation by the prismoidal form yields a very nearly accurate result between 0 and 1.

$$k_1 = \frac{c_0}{6} \left[ 1 + 4e^{-\beta \left(1 - \frac{1}{R}\right)} + e^{-\beta \left(1 - \frac{1}{R}\right)} \right].$$

In the special case of the centrifuge represented diagrammatically in Figs. 1 and 4, the value of  $R$  is 2, and the depth of liquid is 1 centimeter. The expression therefore becomes

$$k_1 = \frac{c_0}{6} [1 + 4e^{-.375\beta} + e^{-.5\beta}];$$

$$c_0 = k_1 \left[ \frac{6}{1 + 4e^{-.375\beta} + e^{-.5\beta}} \right];$$

$$c_x = c_0 \left[ e^{\beta x \left( \frac{x}{2} - 1 \right)} \right]$$

$$c_x = k_1 \left[ \frac{6e^{\beta x \left( \frac{x}{2} - 1 \right)}}{1 + 4e^{-.375\beta} + e^{-.5\beta}} \right].$$

This equation is identical in purpose with the simpler form derived for gravity subsidence.  $k_1$  represents concentration throughout the liquid before subsidence. It is seen that the factor essentially influencing centrifugal subsidence is

$$\beta = m k f_0 g.$$

In the case of the particular centrifuge in mind,  $f_0$  is 40,000. Therefore

$$\beta = 4 \times 10^{\alpha} r^2 d.$$

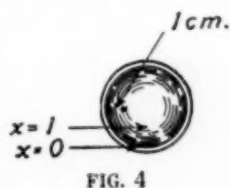
Let us see what happens when we subject a 1 per cent dispersoid to such a force.

$\beta$	$c_1$	$c_2$	$c_0$
1	0.80	0.95	1.36
10	0.06	0.08	6.00
100	10 <sup>-39</sup>	10 <sup>-10</sup>	6.00

The results for equilibrium under centrifugal force are seen to be very similar to those for gravity subsidence. Here again we find that separation is good when  $\beta = 10$  and practically perfect when  $\beta = 100$ .

It should be clearly understood that the formulæ given above are expressions of equilibrium. If a dispersoid whose particles are just visible to the ultra-microscope is subjected to a force of 40,000 times gravity, the particles will be thrown down, provided the force is applied for a sufficient time.

**Computation of Time.**—Many observations in the laboratory under widely varying conditions have led the writer to conclude that precisely the same time is required for each particle of a given dispersoid to move from its position of uniform distribution to the positions of equilibrium. But that this assumption must be correct is evident, not only from observation but from a study of the principles underlying the law of distribution. The particle at  $x = 1$  will move with



greater velocity and a longer distance than the particle at  $x = 0.5$ , but the time will be identical.

The centrifugal force at any point is

$$f_x g = f_0 g \left(1 - \frac{x}{2}\right).$$

The centrifugal force acting upon the particles represented by the concentration,  $k$ , can be expressed by

$$f_x g = k f_0 g \left(1 - \frac{x}{2}\right).$$

Earlier in this paper, it was pointed out that the only considerable force opposing gravity is osmotic pressure.

The osmotic pressure of one particle in unit volume, according to the kinetic theory, is equal to the reciprocal of the constant,  $k$ , that enters into our functions of  $\alpha$  and  $\beta$ . When  $c_x$  represents a concentration by weight percentage, the number of particles in unit volume is

$$\frac{c_x}{m},$$

where  $m$  is the mass of one particle. The number of particles in the volume  $(1-x)$  is

$$\frac{c_x}{m} (1-x).$$

The osmotic pressure,  $P$ , at any point,  $x$ , is therefore

$$P_x = \frac{c_x}{mk} (1-x).$$

Now

$$c_x = k_1 \left[ \frac{6e^{\beta x} \left(\frac{x}{2} - 1\right)}{1 + 4e^{-.375\beta} + e^{-.5\beta}} \right],$$

and if we let

$$\varphi(\beta) = \frac{1 + 4e^{-.375\beta} + e^{-.5\beta}}{6},$$

we can write

$$P_x = k_1 \frac{e^{\beta x} \left(\frac{x}{2} - 1\right)}{mk\varphi(\beta)} (1-x),$$

and since

$$mk = \frac{f_0 g}{\beta},$$

we can write

$$P_x = f_0 g \cdot k_1 \frac{e^{\beta x} \left(\frac{x}{2} - 1\right)}{\beta\varphi(\beta)} (1-x).$$

The actual force  $F_x$  exerted on the particles of a dispersoid can therefore be expressed by

$$F_x = k f_0 g \left[ 1 - \frac{x}{2} - (1-x) \frac{e^{\beta x} \left(\frac{x}{2} - 1\right)}{\beta\varphi(\beta)} \right].$$

When Stokes' law is applied to particles of given mass acted upon by the acceleration of gravity, it is necessary to put

$$\begin{aligned} f &= mg \\ &= \frac{4}{3}\pi r^2 \cdot d \cdot g. \end{aligned}$$

Thus, we have

$$U_x = \frac{2r^2 dg}{9\eta},$$

or for the special case of the centrifuge

$$U_x = \frac{2r^2 dF_x}{9\eta},$$

$$U_x = \theta(\beta) \left[ 1 - \frac{x}{2} - (1-x) \frac{e^{\beta x} \left(\frac{x}{2} - 1\right)}{\beta\varphi(\beta)} \right],$$

where

$$\theta(\beta) = k f_0 g \frac{2r^2 d}{9\eta} = k f_0 g \frac{\beta}{18k\eta\tau_1}.$$

Now velocity, by definition, is the distance traversed in unit time. The time required to traverse the distance  $(1-x)$ , must be equal to

$$\frac{1-x}{U_x}.$$

$$\text{Time } (1-x) = \frac{1-x}{U_x}$$

$$\theta(\beta) \left[ 1 - \frac{x}{2} - (1-x) \frac{e^{\beta x} \left(\frac{x}{2} - 1\right)}{\beta\varphi(\beta)} \right]$$

But, as we pointed out above, the time is not a function of the distance traversed. It is evident that the distance  $(1-x)$  is a function of  $\beta$ , and since the distance to be traversed by any particle depends upon the factor defining the concentration, we may put

$$x = \varphi(\beta) = \frac{1 + 4e^{-.375\beta} + e^{-.5\beta}}{6}.$$

Substituting in the equation for time, we have

$$\text{Time} = \frac{1 - \varphi(\beta)}{U_x}$$

$$\theta(\beta) \left[ 1 - \frac{\varphi(\beta)}{2} - (1 - \varphi(\beta)) \frac{e^{\beta\varphi(\beta)} \left(\frac{\varphi(\beta)}{2} - 1\right)}{\beta\varphi(\beta)} \right]$$

This expression gives the time required for particles to traverse from positions of uniform distribution to positions of equilibrium, in a centrifuge whose radius is 2 and whose depth of liquid is 1. The value is the same for particles of given mass and for liquids of the same value of viscosity.

The following tabulation shows the time,\* according to this formula, required for separation of dispersoids whose dispersion media is water ( $\eta = 0.01$  in c.g.s. units). The density of the particles is taken as 1, and the concentration,  $k$ , is 1 per cent.

Radius		$\beta$	$\theta(\beta)$	Time
$C_m$	$\mu$			
$10^{-8}$	$0.1\mu\mu$	$4 \times 10^{-5}$	$8 \times 10^{-4}$	13 days
$10^{-7}$	$1.0\mu\mu$	4	$8 \times 10^{-4}$	33 hours
$10^{-6}$	$10.0\mu\mu$	$4 \times 10^3$	$8 \times 10^{-4}$	18 minutes
$10^{-5}$	$0.1\mu$	$4 \times 10^6$	$8 \times 10^{-4}$	11 seconds
$10^{-4}$	$1.0\mu$	$4 \times 10^9$	$8 \times 10^{-4}$	.1 second
$10^{-3}$	$10.0\mu$	$4 \times 10^{12}$	$8 \times 10^{-4}$	.001 second

It will be seen that when  $\beta$  is larger than 100, the time is about equal to  $\frac{1}{\theta(\beta)}$  in seconds.

Some objections may be offered to the derivation of our equation for time. Whatever the merits of the case, our equation has two rather unusual characteristics, it is easy to use, and its results, as far as we have observed, will accord with every laboratory measurement.

**Practical Considerations.**—The great point of superiority of the continuous centrifuge over the non-continuous type is the possibility of handling practical volumes of liquid. When the volume is great, the liquid must pass quickly through the rotor and the time during which the centrifugal force can act on the dispersoid is limited. The time in each case is dependent upon the stationary capacity of the rotor. For instance, if we must pass 10 liters of liquid into a given rotor before the level is high enough to overflow (or discharge), it would seem that the time in minutes during which each unit of liquid remains in the rotor will be equal to the rate of flow in liters per minute divided by 10. As

\*It must be remembered that these figures will apply only to the centrifuge exerting a force of 40,000 times gravity and having a radius of 2 centimeters. For any other force or diameter, the proper expressions can be easily derived from the general forms.



a matter of fact, the actual time will be less than this quotient by an interval depending upon the mechanical arrangements of feeding, discharge, etc. Naturally, for any commercial application, a continuous centrifugal will be practical only when it can handle certain required volumes per day. The sizes of particles removed under such restrictions of time will be definitely limited. The capacity of the high speed centrifugal rotor has been limited in practice by strength of materials, but the degree of centrifugal force exerted has not yet reached any such practical limit. It would therefore seem worth while to answer the question: Will any advantage be gained by applying a much greater centrifugal force?

From the tabulation below, it is easy to see a partial answer. These calculations are based on a viscosity of 1. Values of  $f_0$  are taken from  $10^3$  to  $10^7$ .

$r$	$10^3$	$10^4$	$10^5$	$4 \times 10^4$	$10^6$	$10^7$
100 $\mu$	0.5 sec.	5 sec.	50 sec.	12 sec.	5 sec.	50 sec.
10 $\mu$	50 sec.	8 $\frac{1}{2}$ min.	12 sec.	5 sec.	1 $\frac{1}{2}$ hrs.	5 sec.
1 $\mu$	1 $\frac{1}{2}$ hrs.	50 sec.	20 min.	8 $\frac{1}{2}$ min.	50 sec.	8 $\frac{1}{2}$ min.
0.1 $\mu$	.....	.....	.....	.....	.....	.....
10 $\mu\mu$	.....	.....	.....	.....	.....	.....

The values of  $\beta$  for every size of particle with the corresponding centrifugal force shown here are so large that the separation will be complete in every case.

The highest degree of centrifugal force at present commercially applied is 40,000. The lowest practical rate of continuous flow represents in this case a maximum time of about twenty minutes. One per cent suspensions of particles of various densities in liquids of various viscosities can therefore be completely separated by this machine in continuous operation according to the following table:

Size of Particle	Viscosity.
$d = 1.00$	
1.0 $\mu\mu$	.0001
3.0 $\mu\mu$	.001
10.0 $\mu\mu$	.01
30.0 $\mu\mu$	.10
0.1 $\mu$	1.00
0.3 $\mu$	10.00
$d = 0.01$	
10.0 $\mu\mu$	.0001
30.0 $\mu\mu$	.001
0.1 $\mu$	.01
0.3 $\mu$	.10
1.0 $\mu$	1.00
3.0 $\mu$	10.00

Any continuous flow machine can be operated with small separate lots of liquid as an ordinary "bottle" centrifugal. In this way it is possible to remove even smaller particles than shown above.

The determining factors are seen to be viscosity and absolute density. The minimum size of particle is affected by both to the same extent, for we see from the approximate equation for time,

$$\text{time} = \frac{9\eta}{f_0 g k_1 2r^2 d'}$$

that

$$\frac{r_1^2}{r_2^2} = \frac{d_1}{d_2} = \frac{\eta_1}{\eta_2}$$

In practice, one will rarely meet with a case where the density of the medium and the density of the particle are the same to the second decimal place. When such an equality is approximated, it is well to bear in mind the fact that density increases with degree of dispersity. For instance, the density of a globule of water

30 $\mu\mu$  in diameter is 0.5 per cent greater than that of water *en masse*.<sup>17</sup> The density of gold in various forms has been determined<sup>18</sup> as follows:

Molten and compressed.....	19.33
Colloid, ppt. by ferrous sulphate.....	20.71

The question has sometimes been raised as to whether viscosity, the measure of the internal friction of a liquid, can be truly the absolute measure of the resistance to the motion of a particle. One might expect the physical characteristics of the particle to be a potent factor. But it is believed that each colloid particle is enveloped by a permanent film of the dispersion medium, and that whenever the particle is moved, the film will be dragged along.

The accuracy of the figures given for the high power machine has been verified in a number of ways by experimental work. In some cases the value of  $\beta$  has been so small that separation could not be complete and the theoretical concentration of the dispersoid was computed from the equation for  $c_s$ . In cases where  $\beta$  was large, and the liquid was drawn off before a state of equilibrium (i.e., complete separation) had been reached, the concentration was computed from the approximate formula for velocity. When  $\beta$  is large the change in concentration is directly proportional to the time, whereas with a small value of  $\beta$ , the change in concentration will vary as an exponential function of the time. In other words, the time required for the particle to move the first millimeter will be very much less than the time required to move the next millimeter.

One interesting fact, which has been confirmed in practice, is that the particles of a polydispersoid (containing particles of several degrees of dispersity) can be separated accurately in fractions by applying various degrees of force.

For instance, a serum containing finely divided fibrin and corpuscular debris of various degrees of dispersity was subjected to a force of about 1700 times gravity. After the force had been applied fifteen minutes, the serum was perfectly transparent to the naked eye. But a fine porcelain filter was immediately clogged, showing that many particles larger than one micron still remained. The liquid was then whirled again with the same force for two hours. No sedimentation could be seen, and the Berkefeld filtration was equally difficult. A portion of the original serum was now passed through the centrifuge, exerting 40,000 times gravity, in which the time of application was five minutes. The resulting liquid contained only a few particles as large as 0.5 micron and 50 liters could be passed through the filter before clogging.

An emulsion of crude oil and water was found by distillation to contain 9 per cent water. When diluted with gasoline and subjected to the force of 1700 times gravity, 0.9 per cent of water was separated in thirty minutes. The percentage recovery was not increased by extending the time to five hours. The same gasoline mixture was passed through the high power centrifugal at a rate that insured less than a minute for separation. The yield of water was 8.8 per cent.

These two examples might be multiplied indefinitely from the writer's experience. They show in a concrete manner that a separation that is impossible to secure by a lower degree of force, even though the time be extended far beyond the limits of practicability, can often be secured in a very short time by a more powerful machine.

*The Centrifugal Separation of Solutes.*—The experiment of Van Calcar and Lobry de Bruyn, mentioned earlier in this paper, in which three-eighths of the salt

<sup>17</sup>"Handbook of Colloid Chemistry," Wo. Ostwald-Fischer, p. 121.  
<sup>18</sup>G. Rose, Poggendorf's Ann., Vol. LXXIII, 1 (1848).

was crystallized out from a saturated Glauber's salt solution by whirling in a centrifuge, is very interesting in view of the theoretics developed above. With the comparatively low force applied, and the extremely small size of the particles, the value of  $\beta$  would be very small (about  $10^{-4}$ ). The condition of equilibrium of the molecules would therefore show an increase in concentration at the periphery of a very small fraction of a per cent. But this slight increase means *supersaturation*, and supersaturation has been described by P. P. Von Weimarn as a transition between the true molecular dispersoid and the colloid. The value of  $\beta$  would therefore become correspondingly greater, and toward the end of the period (five hours) the effect of agglomeration crystallization would be quite noticeable. This is, therefore, a somewhat more complex problem than a mere centrifugal separation.

The partial separation of certain gases should, according to our calculations, be within the range of possibility. With 40,000 times gravity, the value of  $\beta$  would be small and separation necessarily incomplete, but the time required to reach this state of equilibrium should be reasonably small on account of the very low viscosity. The continuous separation of gases has not yet been actually tried in practice on account of certain mechanical difficulties of separate discharge, but these difficulties will no doubt be overcome. Complete separations of gases cannot be accomplished by any force less than about one hundred million times gravity.

If a mixture of gasoline and kerosene is passed through the rotor exerting 40,000 times gravity, at the slowest practical rate the difference in concentration between the layers at the surface and at the periphery would be much too small for any analytical detection. If the mixture were held in the rotor for three weeks, an equilibrium would be established, but even then the difference in concentration would be in the fourth decimal place. A force of one million times gravity applied for several days should give a quite appreciable subsidence, but a complete separation would require a hundred million times the acceleration of gravity.

**Recapitulation.**—Particles of uniform size suspended in a liquid medium will settle by gravity when the product of their mass in grams by the constant  $3 \times 10^8$  is as large as 10. When the product is less than 10, a partial subsidence will occur until an equilibrium is established.

The only considerable force opposing complete subsidence in this case is osmotic pressure. By the use of a law of distribution, which takes into account the opposing forces, it is possible to compute the concentration at any level when the system is at equilibrium.

When the conditions are such that a particle will not settle completely by gravity, it is possible to cause complete subsidence by the application of centrifugal force when the product of the mass in grams, the force in dynes, and the constant  $3 \times 10^8$  is greater than 10. Here again a partial subsidence with the establishment of an equilibrium will occur when the product is less than 10.

The time required to establish this equilibrium can be computed from a modification of Stokes' law for velocity.

The highest commercial centrifugal force, 40,000 times gravity, can completely remove particles as small as the lower limit of ultra-microscopic observation, but such a separation will require thirty-three hours when the liquid medium is water. For less viscous media the time will be shortened. Smaller particles can never be separated by this force. Particles just too small to be retained by a fine porcelain filter can be removed in a few seconds.

Gases of different densities cannot be completely separated by any force less than about one hundred million times gravity. The same applies to true solutions. But gases should be partially separated by a much lower force within a reasonable time, whereas solutions in liquid media would require several weeks.

Sharples Specialty Co.  
West Chester, Pa.

## The Carbonization of Kelp for the Recovery of Potash

By J. W. Turrentine

The development of an American supply of potash for domestic industries is still unrealized, despite the fact that American resources in this commodity have been known for several years and numerous attempts on a commercial scale have been made to develop the various sources indicated. Efforts have been made to produce potash from feldspar, alunite, potassic brines and kelp. So far, the proper combination of character and accessibility of raw materials and feasibility of methods of handling has not been found that insures any adequate supply of potash for normal conditions of industry and market.

As the result of the antebellum experiments and the greatly stimulated efforts inaugurated since the beginning of the European war, it may be generalized that the giant kelps of the Pacific Coast still offer the best promise of supplying the proper conditions for the establishment of a domestic potash industry. Thus, they occur in sufficient quantity, they are accessible and the product, under normal conditions, may be delivered at the market—the Atlantic seaboard—at a charge for freight that is not prohibitive.

Notwithstanding the numerous experiments made at various times and along various lines looking to the establishment of proper methods for treating kelp, it yet remains to be shown what the costs will be for harvesting the material and ridding it of its high water content. The devices so far designed show great promise of the ultimate solution of the problem of cheap harvesting. This is a mechanical problem which safely may be trusted to American ingenuity.

The leaching of kelp in fresh water for the extraction of the potash salts on the laboratory scale has failed to produce promising results. The destruction or the alteration of the organic constituents of a mash of macerated kelp by fermentation, followed by coagulation of the jelly and filtration, has received some attention and may be susceptible of favorable development. The removal of water from the kelp by pressure methods shows less promise because of the gelatinous condition of kelp and the fact of the potash salts being held in solution in the plant jelly. The rotary kiln, found so adaptable and economical in other industries, has been applied with considerable, though not with unqualified, success, to the drying of kelp. Better results seem promised when the kiln is operated counter-currentwise and the drying is effected in stages. The solution of these problems is of prime importance, since any kelp industry must be based on harvesting, and until that operation is solved no industry can be developed; where ten to six units of the raw material to one of the dry is to be handled as in harvesting, the cost per unit quantity must be maintained at a minimum if that of the dry material is not to be prohibitively large.

With the aid of a climate such as that of southern California, of low rain-fall and that restricted to a few winter months, it should be possible to effect drying economically in the open air, if it were not for the me-



TABLE I—WEIGHT OF WATER EVAPORATED FROM A TON OF PEAT AS ITS WATER CONTENT IS LOWERED, BY 10 PER CENT STAGES, FROM 90 PER CENT TO 10 PER CENT

Percentage of Water in Peat	Total Dry-Peat Content, Pounds	Water Content, Pounds	Water Evaporated for Each 10 per Cent Reduction, Pounds	Residue Left for Each 10 per Cent Reduction, Pounds	Total Amount of Water Evaporated, Pounds
90	200	1,800.0	.....	2,000.0	.....
80	200	800.0	1,000.0	1,000.0	1,000.0
70	200	466.7	333.3	666.7	1,333.3
60	200	300.0	166.7	500.0	1,500.0
50	200	200.0	100.0	400.0	1,600.0
40	200	133.3	66.7	333.3	1,666.7
30	200	85.7	47.6	285.7	1,714.3
20	200	50.0	35.7	250.0	1,750.0
10	200	22.2	27.8	222.2	1,777.8

TABLE II—SUMMARY OF OBSERVATIONS ON DESTRUCTIVE DISTILLATION OF KELP

Item	Percentage	Weight, Grams
Kelp put in retort.....	100%	3000.
Weight of distillate.....	19.90	597.
Weight from Scrubber No. 1—wash water and distillate less wash water used.....	1.74	52.20
Weight from Scrubber No. 2.....	8.40	252.00
Gas.....	16.15	484.50
Weight of charcoal.....	50.10	1503.00
Moisture.....	2.895	86.85
Loss.....	.815	24.45
Total.....	100.00	3000.00

TABLE III—DETAILED OBSERVATIONS MADE OF DISTILLATION OF KELP

Hour	Retort Temp. F.	Meter Temp.	Meter	Cu. Ft. per Hour	Total Cu. Ft.	Remarks
10.20 A.M.	.....	.....	80.873	.....	.....	Started.
30 A.M.	.....	.....	80.878	.....	.005	Bubbles.
11.00 A.M.	.....	.....	81.145	.534	.272	Bubbles, mist.
03 A.M.	.....	.....	81.250	2.100	2.377	Pyrom. moved.
06 A.M.	.....	.....	.....	.....	.....	Distillate.
16 A.M.	.....	.....	81.900	3.000	1.027	Wash water turning green.
25 A.M.	300	80.5	82.750	3.864	1.577	Tested gas: CO <sub>2</sub> present.
26 A.M.	.....	.....	.....	.....	.....	Rapid dist.
33 A.M.	320	.....	83.600	6.075	2.727	Sample 1 not combustible at burners.
39 A.M.	.....	.....	84.175	5.760	3.302	Dist. water in sebr. opaque.
45 A.M.	340	.....	84.800	6.240	3.927	.....
47 A.M.	360	.....	85.100	9.000	4.227	Rapid dist.
53 A.M.	370	8.15	.....	.....	.....	.....
56 A.M.	380	.....	86.100	6.666	5.227	Mist yellow.
12.00 A.M.	390	82.0	86.450	3.715	5.577	Dist. brown, gas.
06 P.M.	410	82.	87.173	3.220	.....	Slightly combust.
09 P.M.	420	.....	.....	.....	.....	Sample 2.
12 P.M.	430	.....	.....	.....	.....	Brown distillate.
15 P.M.	440	.....	88.393	8.100	7.520	Heavy black dist.
20 P.M.	450	83	89.965	9.432	9.092	Sample 3 lights at burners.
25 P.M.	.....	.....	90.234	.....	9.361	Sample 4, burns luminous flame.
35 P.M.	570	.....	.....	.....	.....	.....
37 P.M.	590	.....	90.933	4.836	4.836	Sample 5.
41 P.M.	640	.....	91.223	4.320	.....	Shut off burners.
43 P.M.	670	.....	.....	.....	.....	Exothermic react.
45 P.M.	700	.....	.....	.....	.....	.....
46 P.M.	710	.....	91.480	1.870	.....	Dimin. flow of gas
48 P.M.	725	.....	91.533	1.590	.....	Dimin. flow distil.
49 P.M.	740	84.	91.563	.....	10.69	.....
51 P.M.	760	.....	91.606	1.260	.....	Luminous gas.
54 P.M.	780	.....	.....	.....	.....	.....
58 P.M.	800	.....	91.660	.540	.....	Started burns.
1.05 P.M.	820	.....	.....	.....	.....	No more dist.
07 P.M.	820	85	91.717	.318	10.834	No gas mist.
09 P.M.	820	85	91.763	1.580	10.89	Gas mist.
15 P.M.	830	.....	91.913	1.400	11.04	More gas.
19 P.M.	850	85	92.113	3.000	11.24	.....
24 P.M.	880	85	92.433	.....	11.56	.....
28 P.M.	900	85	92.653	3.300	11.78	Sample 7.
31 P.M.	920	.....	92.853	3.946	11.96	Dim. flow gas.
37 P.M.	950	85.5	93.107	2.436	.....	.....
45 P.M.	980	.....	93.430	2.418	12.557	.....
50 P.M.	1000	.....	93.430	2.418	12.645	Sample 8.
51 P.M.	1000	86	93.607	1.740	12.734	Shut off.
54 P.M.	1010	.....	93.650	.840	12.777	.....
1.05 P.M.	1000	.....	93.717	.360	.....	.....
13 P.M.	970	.....	93.717	.....	.....	.....
33 P.M.	870	.....	93.717	.....	.....	.....
1.13 P.M.	710	.....	.....	.....	.....	.....

mechanical difficulties inevitably to be encountered in handling such large quantities of material as would be necessary in a kelp industry. Kelp, when exposed to circulating air, dries with surprising rapidity. Since the first few percentages of water removed from such material, as illustrated by table I,<sup>1</sup> represents the bulk of the water contained, and since this is removed spon-

TABLE IV—ANALYSIS OF GAS PRODUCED IN DESTRUCTIVE DISTILLATION OF KELP—RESULTS IN PER CENT.

Sample No.	1	3	5	7
CO <sub>2</sub> .....	80.0	70.0	44.0	20.2
Hydrogen.....	2.0	4.0	10.0	3.3
O <sub>2</sub> .....	0.5	1.5	0.8	0.5
CO.....	10.5	8.5	6.7	4.0
H <sub>2</sub> .....	.....	10.6	23.5	32.0
Methane.....	2.88	4.8	4.4	7.6
Nitrogen (by difference).....	4.12	0.6	10.6	32.4
Total.....	100.00	100.00	100.00	100.00
Weight per cu. ft.....	0.106040	0.094	0.074150	0.057600
Cal. value.....	107.45	196.70	352.05	282.54

TABLE V—RESULTS OF THE EXAMINATION OF THE LIQUID PORTION OF THE DISTILLATE OBTAINED IN THE CARBONIZATION OF KELP, AND A COMPARISON OF THIS DISTILLATE WITH THAT OBTAINED FROM DOUGLAS FIR AND OAK UNDER LIKE CONDITIONS

Run No.	Material Used	Quantity Used	Moisture	Total Distillate	Total Distillate Less Moisture	Settled Tar	Charcoal	Gas (By Difference)	Total Time of Distillation	Percentage of Dry Weight of Material		
										Acid as Acetic	Methyl Alcohol 100 per cent	Settled Tar
		Kg.	Gm.	Gm.	Gm.	Gm.	Gm.	Gm.	Hrs.			
a3	Macrocyrtis pyrifera.....	1	116	290	174	...	465	245	...	...	...	...
4	Do.	1	116	295	149	...	470	265	...	...	...	...
5	Do.	1	116	250	134	...	480	270	...	...	...	...
b6	Do.	1	116	255	139	...	485	280	...	...	...	...
7	Do.	1	116	277	161	...	475	248	...	...	...	...
8	Do.	1	116	275	159	...	510	215	...	...	...	...
e9	Do.	1	116	265	149	...	500	235	...	...	...	...
10	Do.	1	116	268	152	...	480	252	...	...	...	...
11	Do.	1	116	262	146	48	485	253	3	0.34	0.14	5.5
12	Do.	1	116	283	167	41	512	305	384	.49	av-	4.4
d13	Do.	1	116	277	161	60	472	251	4	.28	av-	6.8
e14	Do.	1	116	242	126	37	487	271	4	.28	age	4.4
f15	Do.	1	116	217	101	30	515	268	8	...	...	3.4
23	Do.	1	116	277	161	60	485	238	4	.49	.20	6.8
b24	Do.	1	116	305	189	55	460	235	8	.58	.18	6.2
16	Douglas fir (Pseudotsuga taxifolia)	1	190	414	224	60	260	326	3	1.5	.7	7.0
17	Do.	1	190	424	234	80	276	300	254	...	.7	10.0
25	Do.	1	190	450	260	100	300	250	4	1.6	...	12.0
A18	Oak. (Quercus spp.) saw-dust	1	366	550	184	45	200	250	4	2.9	1.1	7.0
19	Do.	1	313	424	111	60	230	346	314	2.6	1.3	9.0
20	Do.	1	313	412	99	37	215	373	454	2.5	1.2	6.0

aRetort kept at red heat 1½ hours.

bHeated rapidly to red heat.

cVery slow distillation at low heat, occupying 12 hours.

dDistillation started 1.20 p.m.; 1.45 p.m., pyrometer 120° C.; 2 p.m., 160° C.; 2.30 p.m., 200° C.; 3.10 p.m., 220° C.; 4.05 p.m., 240° C. (gases slightly combustible); 4.30 p.m., 360° C. (gas burns steadily); 4.50 p.m., 420° C.; 5.10 p.m., 500° C. (no more distillate).

eDistillation started 8.30 a.m.; 9.15 a.m., 160° C. (large watery distillate); 9.30 a.m., 200° C.; 10.35 a.m., 220° C.; 11.15 a.m., 280° C. (gases slightly combustible); 11.30 a.m., 310° C. (distillate oily; gas burns); 12.25 p.m., 500° C.

fDistillation started 9 a.m.; 9.30 a.m., 140° C.; 9.50 a.m., 170° C.; 10.45 a.m., 190° C.; 12 m., 200° C.; 2 p.m., 220° C.; 2.15 p.m., 290° C.; 3.20 p.m., 300° C.; 4.30 p.m., 400° C.

gDistillation started 8.35 a.m.; 9.10 a.m., 160° C.; 11 a.m., 230° C.; 12.45 p.m., 250° C.; 1 p.m., 300° C.; 2.45 p.m., 440° C.; 4.45 p.m., 530° C.

hDistillation started 8.35 a.m.; 9.05 a.m., 170° C. (gases burn); 9.15 a.m., 200° C.; 10.05 a.m., 270° C.; 11.50 a.m., 290° C.; 12.39 p.m., 530° C.

iDistillation started 1.05 p.m.; 1.40 p.m.; 160° C.; 2.40 p.m., 200° C.; 3.20 p.m.; 250° C.; 5.25 p.m.; 500° C.

taneously in the open air, drying floors with plows for turning the material or elaborate racks with mechanical loading and unloading devices suggest themselves. With these disregarded, no methods of artificial drying seem so full of promise as that by means of the direct-heat rotary kiln.

These are matters familiar to those who have studied the problems connected with the potash-from-kelp industry. They are fundamental. And with their proper solution it seems possible that the kelp industry will be established; for dry kelp is a material which appears entirely adaptable to the potash trade. It is a vegetable powder, of a weight of about 50 lb. per cubic foot, which carries about 15 per cent potash (K<sub>2</sub>O), and is comparable to the low-grade salts formerly imported in large quantity.

Since dry ground kelp contains 40 to 70 per cent organic matter, freight charges per unit weight of potash can be greatly reduced by the elimination of the organic matter; that is, by the preparation of high-grade potash salts. If, to accomplish this end, incinera-

<sup>1</sup>Davis, "Uses of Peat," Bureau of Mines, Bul. 16, p. 110 (1911).



tion methods are adopted, the incineration must be effected at a temperature which does not induce fusion of the potash salts (750 deg. C.). Burning in the open air does not fulfill this condition. Destructive distillation suggests itself and seems to offer the desired solution. It already has been shown that valuable by-products are recoverable by destructive distillation when the latter is properly conducted.<sup>2</sup> Thus, from the distillation of 3000 g. kelp in a closed retort, the results summarized in Table II. were obtained.

The details of the experiments and observations are set forth in Table III.

<sup>2</sup>Turrentine, "A note on the Distillation of Kelp," orig. communication, 8th International Congr. Appld. Chem., 15, 313.

The composition of the gas obtained was shown to be as stated in Table IV.

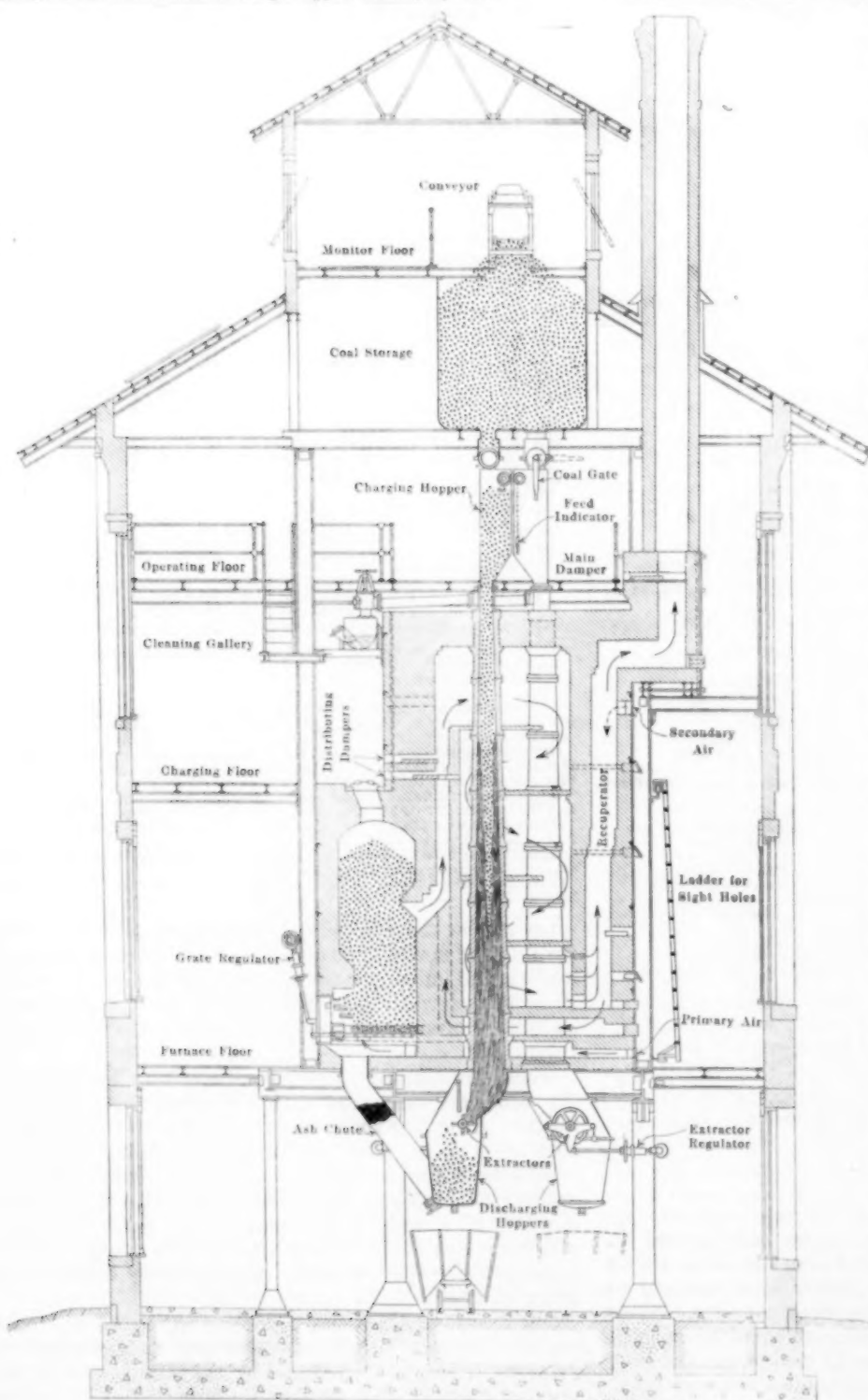
The liquid portion of the distillate obtained when kelp is distilled destructively has been examined by Hoagland.<sup>3</sup> His results are summarized in Table V., from which he concludes that "kelp distillates are of no practical importance." The recovery of ammonia from the evolved gases has not been studied.

The distillation of kelp is analogous, and in a sense similar, to that of coal for the production of illuminating gas and coke. As in the latter so in the former a continuous and automatic process, and one in which heat units are faithfully conserved, is desirable. Therefore, in designing an apparatus to accomplish this purpose

the continuous and automatic gas producers now in successful use in the gas and coke industry would be looked to to supply the apparatus of desired characteristics for the distillation of kelp.

There are on the market vertical gas producers which appear to possess features that would make them especially adaptable to the carbonization of kelp. Such a producer would consist essentially of a vertical retort, 25 ft. in height. This may be made in sections to be mounted upon each other and the sections be of such a graduated size that the retort is larger at bottom than at top, thus insuring an unrestricted movement of the charge in its course downward. At top, this may be joined directly with a magazine or charging hopper, from which the charge will pass automatically downward into the retort. Above, the charging hopper should connect through a gas-tight valve with a storage bin for the prepared charge. The latter may be filled by means of a conveyor. At its lower end the retort should be provided with an automatic discharge or extractor, which passes the coke at graded speed into a gas-tight discharging hopper beneath. Gas would pass out at the top of the retort, and be admitted through a tar trap into the main leading to the condenser house.

The retort should be inclosed in an envelope of brick provided with the proper arrangement of heating flues, regenerative devices and baffles. Heat would be generated in the space surrounding the retort. The air supplied for this combustion should be admitted through various passages constructed in the envelope in such a way that the



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<sup>3</sup>J. Agri. Research, 4, 40 (1915).  
<sup>4</sup>Loc. Cit.

air passing through will be heated in transit. Thus the loss of heat through radiation will be reduced to a small value. Likewise, the primary air, admitted at the bottom of the envelope, should be made to circulate around the bottom of the retort, thus cooling the charcoal contained therein and itself becoming pre-heated. The heat from the waste gases may be recovered by passing them through a tubular boiler situated near the bottom of the stack. Thus the temperature of the gases issuing from the envelope may be reduced to about 300 deg. Fahr.

Such apparatus has been tested for coal and has been found entirely successful with that material. The conditions imposed by the successful distillation of coal, it would appear, are much more severe than those imposed by the distillation of kelp. For the latter, as is evident from Table III, a temperature of 1000° F. is entirely adequate. Probably 750° of temperature would suffice, since considerable heat is evolved by the kelp in undergoing the exothermic reaction, apparently beginning at about 700° F. Coal becomes viscous and swells in coking, while kelp loses volume. Kelp probably would pass readily through a straight retort. It is possible that certain of the distillation products from kelp would condense in the upper portion of the retort, where a low temperature would be maintained, and by admixture with the pulverized kelp interfere with the flow of the charge; in which case the gases could be led out at a hotter part of the retort. In general, it may be said the most serious complications conceivably arising in the carbonization of kelp in the apparatus under discussion appear to be so slight that they readily could be overcome.

The charcoal obtained when kelp is distilled, containing the soluble salts, potassium and sodium chlorides, is porous and easily may be leached. By the application of well-known leaching methods a practically saturated solution of these salts and a charcoal freed therefrom may be obtained. The charcoal could be consumed in an auxiliary gas producer or it might be marketed locally for fuel. The gas evolved from the kelp during distillation, after treatment for the recovery of by-products, would be used for maintaining the temperature around the retort, for which it probably would be more than adequate; and any excess would be applicable to the evaporation of the solution of salts or as an auxiliary fuel in the operation of the kilns. The tar and oils recovered would be useful as fuels if no more profitable application could be found for them.

In short, the system would make possible the complete utilization of any fuels produced and heat units generated. The other by-products obtainable from the distillate could be recovered by methods already well understood, if further experimentation warranted. The large volume of water produced might make the recovery of methyl alcohol and acetic acid impracticable unless they were condensed fractionally. Finally, the crystallization of high-grade salts with the conservation of mother liquors, would make possible the recovery of iodine, for which, after all, there is a certain domestic market.

The cost of such apparatus complete and installed, as constructed for the carbonization of coal, is approximately \$50,000 for a daily capacity of 50 tons coal. This is inclusive of fireproof house. For kelp a cheaper construction would be adequate on account of the greatly reduced temperature maintained and the consequent lack of necessity for the use of high-grade refractory materials. Likewise, on account of the greater ease with which kelp could be carbonized, a greater capacity per retort would be possible with

a consequent reduction in the number of retorts necessary—a value to be determined by experimentation.

The operating expenses would be light. In gas producing plants of large capacity, one man per shift on the operating floor and two, working part time, on the discharging floor, are found to be adequate. The operation of such a plant is almost automatic. Additional expenses would be incurred for the operation of an elevator to deliver the material to be carbonized into the storage bins at the top, and to actuate the discharge rolls at the bottom. Other items to be considered are upkeep and interest on investment.

The principal economy to be expected is the reduction in freight rates, a reduction of from 50 per cent to 75 per cent per unit weight of potash. This item alone would justify the installation. The utilization of the combustible portion of the kelp for the generation of heat to assist in the desiccating operations of the plant would put to use those constituents to which no value would be attached in the market. And finally, other by-products, otherwise lost, would be recoverable if such were found to be justifiable by their value.

Bureau of Soils,  
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## The History and Legal Phases of the Smoke Problem\*

By Ligon Johnson

Only the acute phase of the smelter fume problem is new. The problem itself is older than the Christian era.

While both lead and copper were mined and crudely smelted some three thousand years ago it was not until the Roman occupation of the Iberian Peninsula and the British Isles, which occurred but shortly before the beginning of the Christian era, that there was any evidence of smelting operations on a scale sufficiently large to permit of a fume problem.

Around Huelva, Spain, are found more than 30,000,000 of tons of slag from lead smelting conducted there by the Romans. Pliny tells us that more than 20,000 slaves were employed in the Iberian mines. Extensive mining and smelting by the Romans in England and Wales were coincident with the Iberian proceedings.

These metallurgical operations were upon a sufficiently large scale to produce marked results both upon the surrounding country and the smelter workers, but, as these operations were those of a conqueror upon conquered soil, conducted by slaves, imperial Rome failed to recognize that such a thing as a smoke problem did or could arise, and whatever fume question there may have been at that time remained a question only to those who had no chance to answer any phase of it.

The smelting operations of the Romans extended over about 400 years and little is recorded of lead and copper smelting from that time until the sixteenth century. From the revival beginning at this period up to the present century the growth of smelting has been comparatively gradual. In Great Britain smelting was conducted almost wholly in localities where metallurgical operations were of paramount importance, and the communities which grew up in smelter localities were due to and dependent upon the mines and smelters. This fact had much to do with the comparative freedom of British smelting from burdensome fume litigation and legislation. To a lesser degree these conditions applied to the German operations, which, when coupled with the further fact that most

\*A paper read at the joint meeting of the New York sections of the American Institute of Mining Engineers and the American Electrochemical Society on January 26, 1917.



of the early German operations were to a greater or less degree fiscal workings of Prussia, Saxony and Brunswick-Hanover accounts for the early immunity of German smelters. Topographically and with relation to fertile farm lands, the locations of the British smelters were superior to the German, and ultimately a strong legislative barrier was erected around German operations, particularly in the case of new smelters and under new locations of old plants.

Nowadays German smelting is or at least, prior to war conditions, was under supervision of special officials in the nature of mining police and courts. Before construction of a smelter could be begun application for a permit had to be made, and this application accompanied by general data as to location, character, capacity, height of stack, character of ore to be treated and the like. Where a permit was once granted, it was not revocable, but notwithstanding the permit, if damage was done the smelter could be required to change its methods and to install such appliances as would, so far as possible, prevent injury. Where a permit was refused, no smelter could be built. The refusal of a permit by the Bergpolizei or mining police was not final, but an appeal could be had to the Berg Gericht or Mines Court and finally to the Ober Berg Gericht or Mines Court of Appeals, which was the tribunal of last resort. Incidentally, the Bergpolizei had inquisitorial or supervisor powers not only as to damage to vegetation but also as to the safety of the works and the life and health of the employees.

It is supposed by many that the British Alkali Act was aimed at the smelting industry. This is far from correct. The original acts were directed chiefly at the manufacture of soda, acids, and ammonia. In 1861 the House of Lords provided for an inquiry "into the injuries resulting from noxious vapors evolved from certain manufacturing processes, and into the state of the law relating thereto." While the investigation covered smelting as well as the other enterprises, the resulting act (the Alkali Act of 1863) covered only so-called alkali works and provided for the elimination of not less than 95 per cent of the hydrochloric acid produced in alkali processes. By an amendment of 1874 it was enacted that not more than 1/5 of a grain of acid should be contained in each cubic foot of gas escaping from the works.

The original act was made more comprehensive by amendments and revisions of 1874, 1881 and 1892, but outside of operations under the wet copper process and certain zinc processes it was not until 1906 that smelting generally was included, and, even then, no actual regulation was provided as the act so far as smelting was concerned merely declared that the inspectors under the act "may inquire whether in any works in which sulphide ores are calcined or smelted, means can be adopted at a reasonable expense from preventing the discharge from the furnaces or chimneys of such works into the atmosphere of any noxious or offensive gas evolved in such works, or for rendering such gas when discharged harmless or inoffensive."

No limitation was placed on smelting operations beyond inquiring as to the installation of such remedial methods or appliances as might be installed at a reasonable expense. There was no prohibition as to tonnage or fume strength, and the first report of the inspectors touching on smelting was in the forty-fourth annual report of the department for the year 1907, forty-five years after the passage of the original act, and this embodied no smelter regulations.

Fume controversies and the attendant litigation and legislation are of comparatively recent origin in the United States. Probably the first thing in the way of

legislation in this connection was the ordinance of Oakland, Cal., enacted in 1872, prohibiting the erection or maintenance of any smelter within the corporate limits of that city. Subsequent California legislation was enacted by Contra Costa County limiting fume strength and by the State providing for a smelter commission to investigate the effect of fumes liberated in smelter operations. Efforts were also made to prohibit by legislative enactment the elimination of stack gases containing more than two-tenths of 1 per cent sulphurous content. This proposed legislation, however, was defeated. There has been little legislation either by the States or municipalities of the United States.

Not until heap roasting of ores rich in sulphur was practised on an extensive scale was there any litigation of consequence in this country, and to the condition resulting from heap roasting may be charged no small amount of the prejudice by the farmers against smelters and belief by them of the complete destruction of vegetation, animals and soil vitality.

The basis of this prejudice and belief is easily apparent, even to this day, under a visit to Butte, Shasta County, California; Ducktown, Tenn., and other places where heap roasting was extensively practised.

Under heap roasting, strong fumes, in dense volume, were liberated directly on the surface of the earth. These fumes were heavier than air and cold, being practically of atmospheric temperature. They did not float off in the atmosphere but hugged the ground, rolling along in front of the wind in constant volume until absorbed by the soil and vegetation. There was little diffusion and the radius of fume influence gradually widened with the destruction of each successive barrier of vegetation.

The topography of the country surrounding the roast heaps was, almost without exception, sharply declivitous as the roasting operations were conducted in hilly and mountainous regions. The soils of the regions surrounding the smelter sites were coarse and grainy, requiring the binder of the roots of vegetation and humus to hold the soil particles together.

The destruction of plant life adjacent to heap roasting operations resulted not only in the loss of vegetable life above the soil but of the roots below, and, with every rain, the top soil was eroded until considerable areas were not only made wholly barren, but the top soil washed away and only the decomposing rocks and mica were left. In consequence, there was no soil in which vegetation could find a foothold, even when the fumes were no longer present. It was from this erosion that the belief of the killing of the soil first arose.

From what they could already see, the people around the smelters believe the smelter smoke to be the veritable breath of the Upas tree which destroyed all within its reach, and that this deadly agency was daily reaching farther and farther from the smelter and in and upon the homes and farms surrounding the smelter sites. Litigation was then not only threatened, but instituted. First, the farmers sued the Tennessee and Ducktown companies, and upon the failure of these suits the State of Georgia took up the cudgel. The farmers in Shasta County, California, brought suit against the plants at Kennett and Goram; the Benicia residents and adjacent farmers sued the Selby smelter; the United States, by reason of the ownership of a forest reserve adjoining the Keswick plant, filed its bill against the Mountain Copper Company. Not long after this the Deer Lodge Valley farmers filed their suit against the Washoe Company at Anaconda, and Salt Lake Valley farmers began proceedings against the smelters operating in that valley. The epidemic of smelter litigation was under way.

With these suits developed the early so-called smoke experts. Most of them had been in the heap-roasting communities where the destruction of vegetation and soil erosion had followed the heap roasting. No definite knowledge of fume action was available, and the experts for both plaintiffs and defendants, in the absence of knowledge, began to form pet theories only to be met by pet theories of others directly at variance.

In the good old days of theory an expert could make a casual inspection, or examine a few cross-sections of leaves under a microscope, or make a few comparative tests for the sulphur contents of healthy and unhealthy plants and deliver an epic on what the fumes were or were not doing. The appearance of the country around the old roast heaps and early plants were too big a handicap for the smelters to overcome, and, with the notable exception of the Washoe litigation, in practically every case an injunction followed.

About the time of the institution of those suits the smelters began the abandonment of heap roasting and the development of present-day operations. In this development prior theories expounded by the then experts or testified in the smelter suits had considerable bearing. In one locality everyone was assured that the injury came wholly from the dust particles; in another, from the dust particles as the nucleus for minute drops of acid; in another, from sulphuric acid vapor, which could be seen as a white fume cloud, and practically everything was blamed more than the chief, if not the sole, offender, the  $SO_2$ . In many cases the courts followed these theories in their decrees.

The first development went to long settling flues, smoke houses and dust chambers, cooling and slowing down gases, and taking out so far as possible the solids and vapors. At the Tennessee plants acid making was attempted.

In most of the litigation, under re-adjustment to conform with decrees entered or modifications of these decrees, which modifications as a rule were secured under very substantial cash payments to the farmers, the smelters secured a new lease on life. Several, however, such as the Balakalala plant in California, and the Highland Boy at Salt Lake were completely closed, and passed into history as smelters.

It is interesting to note that not only were the findings of fact in the different cases widely at variance but the conclusions of law in many instances directly in conflict, even in cases in different circuits of the United States courts. For instance, in California and Montana, the United States Court held that the court could consider the balancing of conveniences, that is, that the court, in determining whether or not an injunction would be granted, could weigh the damage which would be done under closing a smelter against the benefits the farmers would receive under an injunction. In the Federal Court in Utah a directly contrary holding was made. In neither law nor facts did the courts coincide.

Some little time was required to make the plant changes to conform with the theories of the experts adopted by the court or the farmers and still further time was required to demonstrate the effectiveness of the changes. Most of the theories failed to pan out and gradually a new crop of complaints and claims that the recent installations did not prevent the damage, began to grow. Some claimants were insistent. Further injunction proceedings were threatened. Plant managers, remembering the results of old litigation, sought to temporize, instead of ascertaining the real facts and meeting them squarely. Some tried to buy peace. Let me say just here that nothing in the Selby report is truer than the statement (page 14) that "the

policy that 'buys off' trouble, as the most expedient commercial method of abating it has been responsible for much of the smelter litigation of this country and the intense ill-feeling that unfortunately exists toward smelters in many smelter communities." It did not take long under this practice for the price of peace to rise to prohibitive figures and a new epidemic of litigation was threatened.

Going back a little beyond this second period of threatened litigation and before the storm clouds of it were yet clearly over the horizon, I think that I may say that the smoke problem was then considered by most plant managers as the least of their troubles. Little or no thought of study was given the question and when it was mentioned it was waved aside. The worst most of them feared was having to judiciously distribute a little peace money.

A number of the smelters, such as the Washoe, Mammoth, Bully Hill, the old Mountain Copper, the proposed Engles smelter and others were either in or adjacent to National Forest Reserves and numerous reports of damage, or threatened damage began to come to the Chief of the Forest Service and Land Office, which reports were in turn referred to the Department of Justice. I was at that time Special Assistant to the Attorney General and these reports were finally referred to me with instructions to prepare and file bills for injunction unless some satisfactory solution could be reached.

While it was probably unknown to the smelters generally at that time, it was never the intention of the Government to close any plant. What it sought was the immediate and serious consideration of the fume problem and active effort to correct harmful conditions where such existed. Only one suit was filed, and that was the suit against the Anaconda plant. In each of the other cases stipulations were entered providing for research work and the installation of the highest types of methods and appliances known to smelting science or else the abatement of the operations producing fumes in harmful quantities until such appliances had been installed and proven elsewhere, upon which they were to be installed at the smelter entering into the stipulation. In the Anaconda case the first scientific commission was agreed upon. We proposed that the smelter operate under the best type of methods and appliances known to science and commercially feasible at the plant and that a commission of John Hays Hammond, Dr. Ricketts and Dr. Holmes, Chief of the Bureau of Mines, be designated as a commission to investigate and prescribe what changes or installations should be made under the agreement.

It was not long after this that the threat of further litigation by the farmers became ominous, and, as I had completed my work with the Department of Justice, so far as smelting matters were concerned, I resigned to become what might be termed smoke or field counsel for the American Smelting & Refining Company.

The first urgent smoke matter in this connection was the Selby litigation, between Benecia residents and the Selby plant, which had been pending a dozen or more years. A flat injunction against the operation of the Selby plant had been granted, which injunction had been fought through the courts and finally approved by the Supreme Court. The case had been through the court of last resort and the injunction was to become effective in a month or six weeks time.

On examining the record I found this suit to be somewhat different from the average smelter suit. Very little claim or injury to vegetation was made. More stress was placed on injury to animals, but the chief ground of complaint was excessive discomfort and al-



leged nausea and illness produced by odors which the witnesses uniformly declared "to smell like rotten eggs" or the sulphur spring of an adjoining county which spring gave off quantities of sulphureted hydrogen.

An examination of the Selby plant disclosed that, regardless of what lead loss there might have been at other times, under changes made or under way no lead loss would occur and therefore no possible source of injury to animals could exist from the plant's operations and that there was no appreciable amount of sulphureted hydrogen given off by or generated in the smelter plant.

In the San Francisco region in the spring and summer months constant trade winds blow and these winds follow a direct line from the Selby smelter to Benicia where most of the complaining witnesses lived. Beyond the Selby plant but also directly in line of the trade winds over the Selby plant was a large oil refinery and asphalt works. The odors complained of by the witnesses all came in the early morning hours, and investigation disclosed that at the times involved in the testimony, the oil and asphalt stills were not capped and were poured at 3 or 4 o'clock in the morning. Here then was a source of odors, which odors traveled on the same winds that carried the Selby smoke. The smelter was guilty of none of the things upon which the injunction was based. Capping the oil and asphalt stills practically disposed of the unpleasant smells.

We notified the county attorney and commissioners of Salano County of these facts and stated that we proposed to decline to observe the injunction prohibiting the operation of the plant. This would mean additional long drawn out and expensive litigation. To obviate such a situation I proposed that the whole matter be referred to a commission of scientists of the highest type, the members to be agreed upon jointly, and the finding to be entered in the Selby case as the finding and decree in that case. Every access to the plant and fullest facility for examination was to be given the commission. After many conferences an agreement along these lines was arrived at and the Selby Commission came into being. The members of the Commission were Dr. J. A. Holmes, Director of the Bureau of Mines; Dr. E. C. Franklin, then director of the Chemical Laboratories of the U. S. Public Health Service at Washington, and Ralph A. Gould, a chemical engineer of San Francisco. With the Selby commission came the first scientific research in smelter fumes along the lines of normal field conditions.

The examination of the Selby Commission extended over a period of about a year and a half and in the end clearly demonstrated that the smelter was doing none of the things found against it in the original decree; and the injunction was vacated.

Shortly after the time the Selby Commission began on its work the murmuring of discontented farmers became much louder. Crops had failed in many localities and wherever this crop shortage was in a smelter locality the smelter was blamed. This was particularly true where prior payments had been made to farmers. Peace was quoted at war prices.

As I before stated, this was very shortly after the Selby Commission began its work. Besides the research just beginning under the Selby Commission, there had not been in the United States or elsewhere, any experiments or research work with smelter fumes or SO<sub>2</sub> under natural field conditions. It is true that Haselhogg and Lindau, Von Schroeder and Reuss, E. Schroeter, Wieler and Hartleb, R. Hartig, Wisliucenus, Freitag, Sorauer, Ramaun, Gerlach, Schnitz-Dumont, Sabachnikoff, Haubner and Stockhardt in Europe, and Haywood and Pierce, and to a limited extent, Ebaugh and Talmage in the

United States had carried on some experiments, but in none of these were normal field conditions approximated. Some fumigations were carried on with leaves or twigs or small parts of plants in bell jars. Others were in hermetically sealed cabinets, or "smoke houses" so constructed that only abnormal conditions could result. The plants used were grown in pots or flats and were grown and kept under conditions that were not normal. The fumes introduced were from burning sulphur or sulphur and alcohol or else concentrated SO<sub>2</sub> let in from a tank or gas burette. The concentrations were unknown, absorption was not considered nor were chemical analysis of the air made. Environmental factors of temperature, humidity, light values, barometric pressure and time element of exposure were not considered. The plants used in the experiments were not grown to maturity nor were their food or crop values ascertained. As a matter of fact at the time these experiments were conducted, no quick and accurate method of air determination was even known to the experimenters. Even had these investigations been conducted with normal plants, under natural field conditions, the lack of knowledge or records of actual fume strengths reaching the plants and of the environmental factors involved, would have rendered the experiments practically valueless.

Some investigators of smelter regions placed much reliance on the sulphur content of plants collected in smelter neighborhoods notwithstanding the fact investigations in the Department of Agriculture have shown that the sulphur content of the same plant may vary as much as 3000 per cent at different times during the season and at the same period the content of the same species of plant will vary markedly, particularly under different percentages of soluble sulphur in the soils, which variance frequently occurs at short distances.

In other words, from farmers and experts the smelters were coming in for full blame for all crop failures and the managements actually did not know whether or not they were doing the damage. From my past experience, I knew that there were numerous plant injuries attributed to smelter smoke which even the most competent expert could not differentiate by mere observation or even microscopic study. Numberless diseases and injuries which were not caused by smelter smoke were pointed out as evidence of smoke damage.

No plant manager knew how much, if any of the damages attributed to the smelter was caused by it, and if there was damage, what operation of the smelter or element of waste had produced the damage. And, too, with the payment of damages communities of smoke farmers grew up. Their idea of farming was to let their places grow up in weeds and collect from the smelter the value of the maximum crops ever produced. Many smelters had paid damages for conditions which, it later developed, were not remotely attributable to smelter operations; but the farmers having once been paid insisted on continued payments. The price of peace was rising above the possible profits of smelter operations.

The time for theorizing had passed. It was necessary that the smelter managements know just what damage, if any, was being done, and if there was damage, how it was done and what was necessary to prevent such damage. Under my urgent recommendation, the first research department to ascertain all this was installed and it, in conjunction with the Selby Commission, blazed the way for real smoke engineering.

This research work involved chemistry, plant pathology, plant physiology, entomology, agronomy, dairy husbandry, and veterinary toxicology and meteorology.

You may wonder why all this was necessary just to find out whether or not the smelter was doing any of the things claimed and why one or two experts familiar

with the appearance of vegetation in smelter regions would not have sufficed.

We have to know first what smelter fume or parts of fume would do damage; how the injury could be caused; what conditions were or could be confused with smoke injury and finally, where damage was or could be done, what steps were necessary to obviate it.

This meant first extensive research work under normal field conditions to ascertain the effect of smelter eliminations upon vegetation and animal life. The time at hand is too short to go into a minute description of this research work. It will probably suffice to say that we found that dust and acid vapors were practically negligible quantities in the fume problem so far as vegetation was concerned. Where damage was done, this could be traced almost if not wholly to sulphur dioxide. The average vegetation can stand fifty times the strength of acid vapor that it can resist where the sulphur is administered in the form of  $\text{SO}_2$ . In the average smelter fume the acid vapor is but a small fraction of the sulphur content, this latter being chiefly confined to sulphur dioxide.

It then became necessary to ascertain the relative resistance of the various plants to sulphur dioxide and the conditions under which injury occurred when the  $\text{SO}_2$  was present in sufficient quantities to do damage.

To develop this fact it became necessary to know exactly how smelter fume acts upon plant life, how it obtains ingress to the plant structures and its effect after its absorption into the plant.

It may not be amiss just here to describe in a few words how this ingress comes about and the results which follow. In the beginning we found that only during the period the plant was in leaf could injury occur. Subjecting a plant to  $\text{SO}_2$  or other fume while not in leaf produced no result. In most instances, and practically without exception in alkaline soils, we found that treating the soil in which the plants grew with sulphur or sulphuric acid, that is, dusting or spreading crude sulphur upon the soil or spraying the soil with acid, resulted in increased crop yield. Sulphur administered in this way was a benefit and not an injury. This conclusion was verified by independent experiments conducted at the University of California in Berkeley, at an experiment station in Oregon, and at the experimental farm of the Anaconda Copper Company.

The question then narrowed down to the effect of smelter fume through leaves and on leaf structure and to present this clearly it is necessary to describe the ordinary leaf and its functions.

All leaves under normal field conditions, in climates such as we have to deal with here, have an epidermis covering both the under and upper leaf structure. This epidermis is impervious to moisture and for all practical purposes, except where stomata or breathing pores occur, is also impervious to gases. These breathing pores are so small that considerable magnification under a microscope is necessary to see them at all. The stomatal openings are entirely too small to admit the minutest suspended water globules or particles of mist or vapor. The stomata at the leaf surface, are faced with two guard cells, that are also covered by practically impervious epidermis, which guard cells, under certain leaf functions, open or close the stomatal chamber. With the guard cells closed the leaf under ordinary conditions presents impervious surfaces to the elements. The inner content of the leaf, or the mesophyll, is composed of palisade cells, collecting cells and sponge parenchyma, in which, under this action of light, starch and sugar are formed and plant food manufactured and supplied the plant.

We next found that under conditions of absolute

closure of the stomata, the plant was a number of times more resistant to  $\text{SO}_2$  than when the stomata were open. Under usual conditions the stomata forms the point of ingress of gas. It followed, therefore, that where an external condition brought about the closure of the stomata it brought about increased resistance, and we finally worked out four factors in chief in fume injury, where sulphur dioxide is present in strengths sufficient at any time to bleach plant life, these factors being light (the stomata are closed during darkness), humidity, temperature, and constant wind direction. The bearing of the latter comes from the fact that injury except from abnormally strong fumes does not come from mere fume puffs but from steady application of  $\text{SO}_2$  for several hours. The other three factors bear upon stomatal opening.

After we had learned the strength of  $\text{SO}_2$  necessary to produce markings upon plant life under field conditions, it became necessary to know whether or not  $\text{SO}_2$  was present in such quantities in the field. It was also necessary to know accurately the wind constancy and direction, the limits of the smoke stream, temperature, humidity and general weather data. Complete installation of standard weather instruments, under the supervision of the local U. S. inspector, was made; and portable laboratories, that is laboratories set up in small automobiles which could follow the smoke stream as well as make general tests, were secured. This was in addition to the fixed air stations maintained. Under the method of air analysis devised by Mr. J. R. Marston and later refined by Mr. A. E. Wells, chief chemist for the Selby Commission, quick and accurate determinations of air samples were possible, and, more important still, these samples would be taken in a few seconds time and, in this way, the constancy or inconstancy of the  $\text{SO}_2$  determined. For the smelter plants we cross-sectioned the several flues at the base of the outlet stacks and the stacks to afford analysis of their contents and installed self-recording thermometers at appropriate points in the flues and stacks for accurate and complete data as to gas temperatures.

With this knowledge the next step was to ascertain with scientific exactness the cause of crop failure or injury where the appearance of external conditions, and often even microscopic cross sections, were seemingly identical with the results of smoke injury, but where our other investigations had developed that smoke injury could not have occurred. There are many diseases, pathological condition and insect injuries, which, both externally and under the microscope, present an appearance practically identical with that of smoke injury. With the completion of this investigation, a survey of the so-called fume zone followed.

It is interesting to note that, in determining the injury of plants by disease, we can establish our diagnosis with scientific exactness not possible in the diagnosis of diseases of human beings or animals. We can establish our diagnosis beyond the possibility of doubt or question. This is done by what is known as the pure culture method. The unhealthy plant is pricked by a needle pointed instrument which in turn is dipped in a special and pure plant jelly and a culture of the disease organism is grown. The disease organism is isolated and reproduced. A healthy plant is then inoculated with this culture and after the disease develops comparison of appearance is made; a second culture is made from the inoculated plant and a second inoculation of a healthy plant follows, and in this way the disease organism is isolated, the disease and appearance reproduced and the diagnosis established beyond a reasonable doubt.

For insect causes, we find and collect the causative insect. As some insect colonies, although numbering



thousands, are so small that the individuals are only observable under very high-powered microscopes, and others work wholly out of sight and beneath the soil, it required a competent entomologist to handle this phase.

It will suffice to say here that we proceeded through the whole list of plant pathology, physiology, entomology, agronomy and dairy husbandry and veterinary toxicology with equal care and certainty. The time is too short to go into detailed explanation of the plan pursued under each.

One other agency in smoke determination has been established with relation to plant life and that is the evidence of so-called guide plants. There are certain plants, such as barley, which are especially susceptible to fume injury and will show bleaching long before the possibility of injury to other plants. Where the guide plants are unmarked, it is self-evident that the trouble with adjoining hardier plants is not smelter smoke.

In the work I have mentioned, we not only secured information valuable to the smelters, but we accomplished actual and beneficial conservation so far as the agricultural resources of the country are concerned.

Scientific farming is not yet widely practiced. Farming methods are often handed down from father to son. Year after year the same crops are planted, often on the same soil. This applies to farming generally and not merely to smelter communities.

In many places not only do the soils become impregnated with disease producing fungus and germs, but the result becomes so wide-spread that no healthy seed can be secured in the community. It often happens that disease conditions may largely be eradicated by proper treatment and planting of the seed and crop cultivation, but these facts as well as that the seed is diseased, are unknown to the farmer.

I recall a case where enormous claims for loss of potato crops were filed against a smelter. Upon investigation we found both the potatoes and the soil of the region highly infected with two of the most disastrous of potato troubles, rhizactonia and fusarium. A subsequent search of the seed stores in the community failed to disclose a single healthy sample of seed. Naturally the potato crops were failures. The sad part of it was that there was no need of this loss. Healthy seed procured elsewhere and planted in soil where potatoes had not been grown for several years, would have given the old time record crops, and proper treatment of the local seed would have produced infinitely larger returns.

Most of the diseases and insect conditions of agriculture communities are largely preventable and for those that are not, steps may be taken to greatly reduce the harmful effect. Much of the troubles can be directly pointed out to the farmer, and the means of eradicating or lessening these troubles can be shown him. This, of course, involves the demonstration that the unhealthy crop conditions are not attributable to the smelter.

The farmer who follows the suggestions for eradicating unhealthy conditions soon finds his crops exceeding those of his neighbors who have failed to observe the same precautions. In this way, gradually it is true, the influences of the smelter's experts is felt, a friendlier feeling is engendered, agricultural conditions are improved and actual conservation accomplished.

This brings us down to one final theory which in some places is still firmly entrenched and threatens possible trouble. I refer to visual clearance and the belief that anything seen coming from the smelter stack should be labeled with a skull and cross bones. Our investigations showed that the visible fume was little to be

feared as an actual instrument of damage to vegetation. Under former operations where a good part of the lead content of the ore was blown up the stack and where heavy arsenic fumes were given off, this metallic loss had some connection with certain animal troubles, but had little effect on vegetation. Where this character of loss was not involved, the visible fume was and is chiefly dangerous from the psychological point of view. By this I mean that most farmers (and many will be honest in their belief) will assert, so long as there are visible stack eliminations, that every crop failure is due to the smelter fumes. Therefore, to meet this, it will be advantageous to obtain the greatest possible clearance up to the point—and I want to stress this especially—where the efforts to secure visual clearance renders the smelter operations more liable to do actual damage. Beyond this point it is better to try to educate the farmer and get rid of the belief. Just because a farmer sees dense volumes of soft coal smoke pouring from the stacks of manufacturing plants, business houses, engines and the like, he feels no uneasiness about his crop. There is little if any more danger to crops in the visible smelter smoke. And, too, even where the belief of damage is deep seated, mere belief can be met by facts, but actual damage cannot be dodged because the smelter management seeks to meet some psychological condition; for mere belief won't last long in the face of actual damage.

Get clearance first for actual recoveries, and loss of toxic solids, and next, so far as possible, to meet any physiological conditions, but do not place visual clearance above the likelihood of actual damage.

Our investigation and the temperature curves we have worked out have shown us that where there is fume injury the answer to the problem is in hot gases, gas dilution, and high stacks. Anything that cools or slows down the gases or results in their liberation at low altitudes invites, to say the least, the probability of actual damage. Promising a community complete visual clearance or encouraging the belief that visual clearance means impossibility of damage is borrowing trouble for the future, for such a clearance, unless I am mistaken, means low gas temperatures.

These smoke problems are bringing to the front a new specialist, the smoke engineer. The far-sighted smelter management, under existing conditions, for all new construction, and even alterations and additions to old plants, will find this new expert a necessity rather than a luxury. The best way to take care of trouble is, not to stop it after it has started, but to prevent its starting. Many of the smelters realize this and those that do, in the matter of fume problem, welcome the coming of the smoke engineer and specialist.

### Spring Meeting of American Chemical Society

The condensed general program of the spring meeting of the American Chemical Society, to be held in Kansas City, Mo., April 10-14, is as follows:

Tuesday night, April 10—Council meeting.

Wednesday morning, April 11—Opening session.

Wednesday afternoon, April 11—Opening session, continued, or section meetings.

Wednesday night, April 11—Smoker.

Thursday morning, April 12—Section meetings.

Thursday afternoon, April 12—Section meetings.

Thursday night, April 12—Banquet, or open.

Friday morning, April 13—Section meetings.

Friday afternoon, April 13—Excursions.

Friday night, April 13—Banquet, or open.

Saturday morning, April 14—Excursions.

## Potash as a Byproduct from the Blast Furnace\*

By R. J. Wysor

Since the outbreak of the European war, few problems of raw-material supply have commanded more nation-wide attention than potash. It is well known that before the war the domestic production of potash was an insignificant percentage of the imports. The average annual importation of raw potash salts for several years prior to 1914 was slightly over 300,000 net tons, and of other potash manure salts about 700,000 tons. The sudden and almost total cessation of these imports created a unique and stringent situation. Methods of recovering potash from feldspar and other native mineral sources, and reclamation from hitherto waste products, have received a marked impetus. Soaring prices have been a keen incentive to research and industrial development.

Although several brief articles concerning the possibilities of potash recovery have appeared in recent trade journals, it still may be a matter of surprise to the average technical mind that potash might be reclaimed as a profitable byproduct in the manufacture of pig iron. At the present time it may be of special interest to present some data, largely of a technical nature, on this subject.

A fairly thorough search of the literature reveals a number of articles concerning salts of the alkali metals in the blast furnace. Many of them deal with the theoretical rôle of alkali cyanides in the furnace. Several discuss the probable effect of the alkalies on the furnace brickwork. Two or three ambitious patents have been granted for reclaiming potash or other products from blast-furnace gas. However, we have heard nothing further as to the practical application of these patents. Little literature of importance has appeared during the last 10 years concerning alkalies in blast-furnace practice, and I have discovered no record whatsoever as to the actual sale or commercial disposal of flue dust for its potash content until this was inaugurated in our plant at Bethlehem.

About 4 years ago, in the course of investigating blast-furnace stove efficiencies, I analyzed the fine, yellowish fume of which a considerable quantity was removed from the bottom of the stove checkerwork. The sample was found to contain, among other constituents, about 15 per cent water-soluble potash, which was somewhat surprising. This induced an investigation, which showed that considerable quantities of this material, hitherto a waste product, could be recovered from our stoves and gas-fired boiler settings. A search for a market was made without immediately satisfactory results. One fertilizer dealer claimed that the alumina content was too high. Others wished to make practical tests, and we furnished large samples for a full season's demonstration, satisfactory results being reported. But the pre-war-time prices offered hardly seemed to justify the trouble of reclaiming the dust.

With the beginning of the war and the subsequent spectacular rise in the potash market, conditions were changed. Knowing just what dust to recover, reclamation was immediately commenced, a satisfactory contract was negotiated, and this company has been disposing of the dust at a good profit ever since.

### OCCURRENCE OF ALKALIES IN RAW MATERIALS

Potash doubtless occurs almost entirely in some form of feldspar or clay in all blast furnace raw materials.

At Bethlehem we receive ores from many quarters of the globe, and an attempt was made to discover whether the relative percentages of alkalies in the various ores bear any special relation to the source, mineralogical nature, or to the silica and alumina contents of these ores. No striking relation was discovered, except that the manganese ores, from widely separated sources, were found to contain relatively high percentages of potash as compared to most iron ores. Iron ores of this country, including those of the South, containing upward of 1 per cent potash, or over, seem to be restricted to small areas.

The percentage of potash in different varieties of limestone and dolomite used as flux varies greatly, largely on account of intermixed clay, and may be surprisingly high. Per unit weight, the potash content of our flux charge at Bethlehem is considerably higher than in either the average ore or coke charge.

Few data are available on the alkali content of coke from different localities. Various standard Connells-ville cokes which we have examined have shown a much lower alkali content than our local byproduct coke made from West Virginia coals.

It may be a matter of some surprise to note that, with one exception, the soda contents of all the iron ores, the coke and the flux, subsequently listed, are much higher than the potash contents.

In the course of this investigation, samples of slag and flue dust from all of our Bethlehem furnaces were collected continuously over a period of 4 weeks during June and July, 1916, and analyzed for the alkalies.

The ratio of potash to soda is considerably higher in the coke than in the ore and stone. For each ton of pig iron produced, nearly 60 lb. of the alkali oxides are charged, constituents which certainly are of considerable importance in the working of the furnace, yet of which relatively little cognizance has been taken in the past.

However, due largely to the relatively high percentage of alkalies in our fuel and flux, and on account of the considerable quantity of alkali-bearing flue dust removed, it is my opinion that more potash and soda are charged into our blast furnaces, per unit of iron produced, than in any other large plant in this country.

### ACTION OF ALKALIES WITHIN THE FURNACE

It is probable that a considerable part of the potash and soda charged into a blast furnace is evolved from the top by direct volatilization or heat decomposition, though alteration by chemical reaction of the alkaline salts or compounds liberated may occur before they have left the furnace. It is certain that a large part of the alkalies are carried down into the hotter zones of the furnace and are converted into cyanides by reaction with red hot carbon. Some investigators have attributed an appreciable part of their ore reduction to the action of cyanides, inferring that after being oxidized and driven to the cooler upper portion of the furnace they condense and are again carried down into the reducing zone. Whatever the action, it is self-evident that eventually the same amount of alkalies must be carried out of a furnace that is charged, and this takes place through the following avenues of escape:

1. *In Chemical Combination in the Slag.*—The literature is almost barren with reference to the presence of alkalies in blast-furnace slag. On account of the readiness with which the compounds of these metals are sublimed and carried out of the furnace with the gas, it might be inferred that only a negligible proportion would be found in the slag. This is far from the truth. Average samples from each of six furnaces in

\*Abstract of a paper to be presented at the New York meeting, February, 1917, of the American Institute of Mining Engineers. The author is superintendent of blast furnaces at the Bethlehem Steel Company.



operation for two bi-weekly periods were carefully analyzed for potash and soda.

The calculated weight of slag produced during this period was about 0.52 ton per ton of pig iron produced, which would account for a loss of alkalis as follows:

	Pounds	Per Cent of Total Charged
Average potash in slag per ton pig.....	4.5	26
Average soda in slag per ton pig.....	6.3	17

The percentage loss of the two alkalis in the slag is seen to be about the same, being somewhat greater in the case of potash.

2. *As Cyanide or Other Volatile or Inflammable Compound through the Iron and Cinder Notches.*—Part of the fume arising from the molten iron, and especially from the slag running from the furnace, is undoubtedly alkali compounds. I have often noticed a peculiar lavender or violet flame around the iron and cinder notches during casting or flushing, which I believe is due to some alkali salt or salts burning. The fume arising from the iron and cinder runners certainly contains a considerable percentage of alkalis.

3. *By Liquid Exudation or Decomposition from Gas Around the Tuyères, Coolers, Mantel and Cooling Plates.*—Occasionally while removing a tuyère or cooler, a stream of liquid "cyanide," resembling water, will run out of the furnace, or can be seen exuding from the lining.

When cooling plates above the mantel burn out, due to the failure of the water supply, and a little gassing has commenced, beautiful white and yellow or yellowish-red crystals often build up around the apertures.

Liquid material sometimes also exudes from around the mantel plates and solidifies in heavy columns or stalactitic masses on the shell. This substance, containing some cyanides, is highly deliquescent.

4. *By Combination with the Brickwork or as an Accretion in the Form of Cyanide, Etc., and Removal when the Furnace is Blown Out.*—This is, of course, a relatively small but very interesting part of the alkalis charged during a campaign. Several authors have suggested that destruction of brickwork in certain stacks, particularly in the middle and upper zones, was due to action of alkalis. However, so far as chemical action is concerned, rather more importance has been attributed to reaction between carbon monoxide and iron oxide spots in the brickwork, with subsequent disruption of the brick mass. The destruction of linings is doubtless hastened somewhat in furnaces in which the burdens are relatively rich in alkalis.

The average sample of the hearth brick used in one furnace lining at Bethlehem by careful analysis showed a total alkali content of 1.60 per cent. After blowing out, a sample was secured of a number of these brick near the bottom of the hearth, appreciably discolored when broken, which showed a total alkali content of 3.36 per cent. Higher up in the lining the alkali enrichment is greater. Samples of pure white, mixed cyanides, etc., have been recovered in various plants from protected crevices in the brickwork, around the boshes after furnace campaigns, and a great deal more would be found if the contents of the stack could be removed dry.

The presence of ammonia around furnaces directly after being blown out is, of course, well known. Sometimes enormous quantities are evolved, the odor being apparent until the entire lining is removed and the hearth cleaned out. The chief source of ammonia gas is undoubtedly the alkali cyanides and ammonium chloride which decompose in the hot water or water vapor introduced to cool down the furnace. In passing, it is

interesting to recall that a very small part of the cyanide produced in a furnace is fixed and carried down into the salamander in the form of the peculiar compound, titanium cyano-nitride. The excess accumulation of alkalis in the brickwork, or in salt deposits in a furnace lining after a campaign, may literally amount to several tons.

5. *By Evolution in the Gas.*—The heavy flue dust which is carried out of the top of the furnace by the gas current, of course, contains approximately its normal percentage of alkalis. The finer portions of ore, stone and coke dust doubtless average somewhat higher in alkali content than the entire materials as charged. All the remaining potash and soda in the burden, not previously accounted for, is sublimed and passes out in the form of various salts or compounds as a fine fume.

Various investigators have studied blast-furnace flue dust with special reference to its potash content, but almost entirely from a scientific standpoint. It is true that at least one American and one foreign patent have been issued for the recovery of potash in flue dust, but no evidence has been found that any practical application has thus far been made. And, so far as can be ascertained, the first commercial disposition of the flue dust as a fertilizer was made by us at Bethlehem somewhat more than two years ago.

The progress of the alkalis in our practice will now be traced from the furnace top, through the gas mains, washers, stoves, boilers and stacks.

#### ALKALIES IN DRY DUST BETWEEN FURNACE AND WASHER

Besides the dust carried out by the relatively small amount of gas escaping from the furnace top, and the insignificant quantity permanently deposited along the mains, all the dust under the above heading is removed from the dust catcher. Representative bi-weekly samples of the dust-catcher flue dust were taken over the same period during which the slag samples were secured and analyzed, showing the following averages:

	Total		Water Soluble	
	K <sub>2</sub> O	Na <sub>2</sub> O	K <sub>2</sub> O	Na <sub>2</sub> O
Average for all furnaces, first period.....	0.53	1.16	0.31	0.30
Average for all furnaces, second period.....	0.70	1.42	0.25	0.20
Average for all furnaces, both periods.....	0.61	1.29	0.28	0.25
Ratio average total potash to average total soda.....	1:2.1			
Ratio average water-sol. potash to average water-sol. soda.....	1:0.9			
Ratio average water-sol. potash to average total potash.....	1:2.2			
Ratio average water-sol. soda to average total soda.....	1:5.2			

A much greater percentage of the total potash than of soda is present in a water-soluble condition.

The entire absence of cyanides in this dust, and even in concentrated alkaline samples, recovered further along in the gas system, furnishes further corroboration of the fact that in normal operation practically no cyanide is carried in the gas current from the furnace top. That it may be an occasional constituent of flue dust, because of abnormal furnace conditions, is evidenced by several records at hand.

Tests made at different times show that our dust catchers remove an average of about 100 lb. of dust per ton of pig iron produced. Using the average percentage of alkalis in flue dust for the four weeks, we find:

	Pounds	Per Cent of Total Charged
Average potash in flue dust from dust catcher per ton of pig.....	0.6	1.8
Average soda in flue dust from dust catcher per ton of pig.....	1.3	2.3

#### EFFECT OF PRIMARY WASHERS ON ALKALIES IN DUST

At Bethlehem we have in service a type of tower spray washer, in common use in this country. All of the gas for stoves and boilers, as well as for gas engines, is

washed, except from one furnace. Naturally, it would be thought that practically all of the alkaline material in the dust, most of it readily soluble in water, would be removed in the wet washers. The bulk of it is washed out, but it is a remarkable fact that much of the water-soluble alkalies remains in the gas current after leaving the washers. Calculations based on alkalies charged into the furnaces, lost in the slag, dust catcher, stack gases, etc., and recovered from the stoves and boiler settings, indicate that about 21 per cent of the total potash (apparently only about 5 per cent of the soda) entering the primary washers, passes through them. The explanation for this fact is that the particles of fume are in such an exceedingly fine state of division that they escape contact with the relatively large drops of water. In my opinion, any washer which will successfully clean blast-furnace gas rich in this fume must employ spray nozzles or other devices which will discharge water in a fine mist, thus insuring intimacy of contact between dust and water particles. Our washers at present perform the function of selective precipitation, eliminating the relatively coarse and heavy iron ore and coke particles, while delivering the lighter particles of dust and fume into the primary clean gas main.

#### ALKALIES IN DIRT FROM PRIMARY CLEAN GAS MAINS

The mains carrying gas from the primary washers to stoves, boilers and secondary gas-cleaning plant accumulate dirt or mud gradually, and are washed out at intervals of every two or three months. This dirt is black when wet but after being dried is dark gray, and after the fine particles of coke dust are burned out, it is of a light-gray or reddish-white color. Sometimes drippings from a clean-out door along a gas main will form beautiful long stalactites. This material is in general appearance similar to the stalactites mentioned as exuding from the furnace shell. A typical sample was found to have the following proximate composition:  $\text{SiO}_2$ , 0.28 per cent;  $\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$ , 0.68;  $\text{CaO}$ , trace;  $\text{MgO}$ , 0.25;  $\text{K}_2\text{O}$ , 44.63;  $\text{Na}_2\text{O}$ , 7.13;  $\text{Cl}$ , 37.22;  $\text{CO}_2$ , 7.78;  $\text{CN}$ , trace;  $\text{NH}_3$ , none. The almost utter absence of cyanide in this concentrated alkaline material is worthy of note.

#### ALKALINE DUST IN STOVES AND BOILER SETTINGS

The dark, wet dirt carried into the stoves, which at our plant are of the three-pass type, is dead burned, of course, the combustible or volatile constituents, etc., being expelled. The residue, in the form of an impalpable powder, collects to some extent in the bottom of the combustion chambers, a relatively large amount gathers in the bottom of the second and third pass checkers while the largest portion is carried out in the stack gases.

In the combustion chambers the dust frits together in friable masses. The accumulation is not great, the wells being cleaned out at intervals of about two months. However, the action on the brickwork is serious. All over the inner surface of the combustion-chamber walls, the lower surface of the dome, and the second pass checkers for a depth of several feet, a deep-green glaze is formed, or the brickwork is honeycombed. Along the fire-clay joints between the brick, the action is especially marked. Stove brick of a high silica content invariably glaze or slag, whereas those with a lower percentage of silica are more readily honeycombed or excoriated by the alkaline fume.

The thermal efficiency of the stoves is reduced, of course, by this glazing, erosive and honeycombing action, though repairs seldom have to be made during a furnace campaign.

The fine, light-colored dust, which accumulates in the

bottom of the second and third passes, is the material of commercial interest to us. It is cleaned out at intervals of three or four months. A typical pile of this dust freshly removed through one of the stove clean-out doors is shown in Fig. 1.

In the boiler houses the dust accumulates and is recovered every few days from the combustion chambers, from the passes and occasionally from the horizontal flues leading to the stacks. It becomes increasingly fine and richer in potash in its progress. The boiler tubes, of course, become coated with the dust and must be blown off with a steam or air lance.

The white fume continuously issuing from the top of the stove and boiler-house stacks is approximately of the same composition as that reclaimed in the last passes.

#### PROPERTIES AND QUANTITY OF ALKALINE DUST RECOVERED FOR SALE

As previously indicated, the dust recovered from the combustion chambers and stoves and boilers is a light, friable sinter, whereas the larger amount reclaimed from the passes and flues is in an exceedingly fine state of division. When drawn out of the stoves or boiler settings hot, it runs almost like quicksilver, but it absorbs a certain amount of water readily, and becomes somewhat clammy and heavy.

Sieve tests were attempted. By careful sifting, it was found that all of the alkaline dust would pass a 300-mesh sieve. It is a true fume. The small residues remaining on the various sieves, when examined under a magnifying glass, were found to be chiefly eroded brickwork.

Specific-gravity tests, also, cannot be made with accuracy. The lightest dust, without compression, will run well below 0.50 specific gravity. Samples taken from the checkerwork of five different stoves, and showing potash contents of 10 to 14 per cent, when tramped down gently in large glass measuring cylinders, showed the following varying densities: 0.68, 0.69, 0.73, 0.89, 0.96.

Knowing the exceedingly fine state of division and smooth, spherical nature of the ultimate particles in this fine fume, there is little cause for wonder that so much of it passes through a creditable wet washer untouched.

The water-soluble potash content of the dry, ignited dust, recovered from the stoves and boilers, having previously passed through the wet washers, will vary from about 5 to 20 per cent. Our practice has been to store the material in a large bin, capable of holding two or three carloads. The commercial recovery, begun in

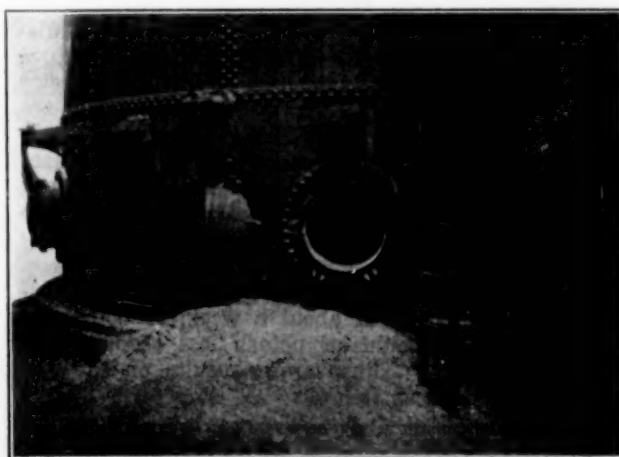


FIG. 1—PILE OF DUST FROM STOVE



1914, was not thoroughly systematized until the ensuing spring. Thirty-six carloads, containing 1,073.5 tons with 106.3 tons of water-soluble potash, of alkaline dust shipped in the period of fifteen months, April 1, 1915, to July 1, 1916. This represents all the dust removed from stoves and boilers serving four 500-ton furnaces, which could be recovered in normal operation, without special effort. Instead of being dumped into a dirt car, the dust was simply emptied into a central bin. A number of other carloads of lower-grade material, contaminated by flue dust from unwashed gas, were also shipped from the other furnaces, but are not included in the above amount.

No readily volatile constituents, such as may occur in the unignited dust, of course, are to be found after roasting.

In general, the ratio of water-soluble to total potash is higher than in the case of soda.

The preponderance of both total and water-soluble potash over the corresponding soda determinations is impressive, showing the much greater removal of the latter in the wet washers, or its greater volatility, in which case it would be carried out of the stacks in greater quantity.

If we assume that the total soda content in the stove and boiler dust averages 40 per cent of the total potash content, and the water-soluble potash 80 per cent of the total potash, as appears reasonable from the foregoing analyses, then the total alkalies recovered in the fertilizer material in the above period is found to be:

Total potash .....	118 gross tons
Total soda .....	47 gross tons

Calculating on the basis of pig iron produced in the above fifteen-month period, the alkalies recovered in terms of total charged are as follows:

	Per Ton Pig. Pounds	Per Cent of Total Charged
Total potash recovered in fertilizer material 0.3		1.3
Total soda recovered in fertilizer material.. 0.1		0.3

The amount of potash recovered, though of considerable tonnage and value, is an insignificant percentage of the total charged.

At furnace plants using burdens rich in potash where the gas is unwashed, a considerable recovery still can be effected, though some precautions must be taken to avoid the coarser, raw flue dust. Several other furnace plants in the East, acting upon our suggestion, made directly or indirectly, have been recovering potash-bearing flue dust during the last year and a half.

#### ALKALINE DUST ESCAPING FROM STOVE AND BOILER-HOUSE STACKS

Some idea of the relative amount of alkalies discharged per unit value of gas from blast-furnace stove and boiler stacks may be gained by observing the color and depth of the fume. Dirty gas, of course, will obscure greatly the fine, white, alkaline fume. In Western practice, with washed gas, the escaping stack gases show a thin, white color. In our practice the stack fume is much heavier. At the plants in the Lebanon district, using Cornwall ore, the fume is relatively very dense.

About two years ago we made a series of tests, extending over a period of about a week, to determine the approximate amount of potash lost through our boiler-house stacks. The average results on the two largest stacks were as follows:

Average amount of dust per cubic foot, flue gas (62 deg. and 30 sec.), grains .....	0.12
Average percentage water-soluble $K_2O$ in this dust.....	14.70
Average percentage total .....	18.60

Knowing the average volume of flue gas discharged, and the average volume of fuel gas consumed by the boiler houses, and produced by the furnaces, we can calculate roughly the weight of potash lost through the boiler-house stacks per ton of pig iron.

No actual tests have been made on potash losses from the stove stacks, but they should be slightly less per unit volume of fuel gas burned on account of the greater baffling action of the stove brickwork. We will assume these losses per unit volume of fuel gas to be 90 per cent as great as from the boiler-house stacks. The soda-potash ratios will be assumed to be the same as in the average stove dust.

	Per Ton Pig. Pounds	Per Cent of Total Charged
Total potash lost in boiler-house and stove-stack gases .....	2.6	11.2
Water-sol. potash lost in boiler-house and stove-stack gases .....	2.2	..
Total soda lost in boiler-house and stove-stack gases .....	1.1	2.8

#### ALKALIES LOST IN SECONDARY WET WASHER

A considerable percentage of the primary cleaned gas at Bethlehem is further cleaned in Theisen scrubbers for gas-engine use. Practically all of the fine dirt is thus removed, the small amount passing through the fine gas main and into the combustion chambers of the engines, having practically no cutting or scouring action, even when as much as 0.03 grain per cubic foot of gas is present. Samples of this dust, recovered near the engines, were of a smooth talcy consistency. The composition is similar to the finest stove dust.

The amount of alkalies recovered in the secondary scrubbers, per unit volume of gas, is practically the same as the sum of the alkalies precipitated in the stoves (or boiler passes) and escaping through the stacks. Knowing the gas-engine consumption of fuel gas, the alkalies removed in the wash water, calculated on the same basis as for the stoves and boilers, is readily found to be as follows:

	Per Ton Pig. Pounds	Per Cent of Total Charged
Total potash lost in secondary scrubbers.. 0.5		2.2
Total soda lost in secondary scrubbers.... 0.2		0.6

#### BALANCE SHEET FOR ALKALIES CHARGED, REMOVED AND LOST

Bearing in mind the extremely difficult nature of the problem, and the necessity for including a number of estimates, I will endeavor to summarize a rough balance sheet for alkalies, charged and produced in our average blast-furnace practice. Further, it will be remembered that this balance sheet is approximately representative of our local conditions only.

Apparently there is a greater loss of soda than of potash in the primary washer. This may be due to the greater solubility of the sodium salts, or to the larger size or possibly to the difference in contour of the fume particles of sodium compounds.

#### METHODS FOR FURTHER RECOVERY OF POTASH

In our present gas-cleaning practice, it appears that there is a loss in the primary washers alone of over half of the total potash charged, or about 12 lb. per ton of pig iron produced, though, as mentioned later, this amount is probably a little high. The amount recovered, while appreciable, and representing almost clear profit, is seen to be an insignificant part of the total, less than  $\frac{1}{2}$  per cent. Potash lost in the slag, around the shell and from the top of the furnace, for all practical purposes, is lost beyond recovery. The greater part of the alkali content of the flue dust removed from the dust catchers could be recovered by leaching in water,

TABLE VI—BALANCE SHEET FOR ALKALIES CHARGED, REMOVED AND LOST

	POTASH (K <sub>2</sub> O)		SODA (Na <sub>2</sub> O)	
	Pounds per Ton Pig Iron Produced	Per Cent Total Charged	Pounds per Ton Pig Iron Produced	Per Cent Total Charged
Total charged.....	22.4	.....	35.6	.....
Lost in slag.....	4.5	20.0	6.3	17.0
Lost in fume, etc., from iron and cinder notches, and shell (est. 10 per cent of slag loss).....	0.4	2.0	0.6	1.7
Lost in gases from top of furnace (est. 5 per cent total gas losses).....	0.9	3.9	1.4	4.1
Lost by combination with brick-work of furnace, stoves, etc. ....	negligible		negligible	
Recovered in dust-catcher dust.....	0.6	2.7	1.3	3.6
Lost in primary washers (est. by difference).....	12.5	55.9	24.6	69.5
Lost in secondary washers.....	0.5	2.2	0.2	0.6
Recovered in stove and boiler passes.....	0.3	1.3	0.1	0.3
Wastage from mains, stoves and boilers (est. 30 per cent of quantity recovered).....	0.1	0.4		0.1
Lost in stack gases.....	2.6	11.2	1.1	3.1
Total.....	22.4	100.0	35.6	100.0

but the percentage is too low to justify reclamation in this way. However, the potash now lost in wet washers and from stove and boiler-house stacks offers a legitimate and inviting field for its recovery. According to our balance sheet, it appears that about two-thirds of the total potash charged is now lost in the wash water and stack gases, or about 15 lb. per ton of pig iron produced.

About two years ago we obtained estimates for cost of recovery of the flue dust from several of our large boiler-house stacks, both by filtering and electrical precipitation methods; but the relatively high cost and the uncertainty in the potash market, deterred an actual installation. Later it was realized that the principal loss of potash was not from the stacks.

At intervals during the course of the last year or more, we have had in operation an experimental Cottrell electric dust precipitator, connected to the raw gas main leaving one of the dust catchers. It is not my purpose in this paper to discuss the operation of the unit, except to state that practically all the dust and fume entering the treater could be precipitated successfully. The color of the dust recovered varied from a light to a dark gray. Several samples were analyzed and showed a potash content of about 10 per cent. The total dust leaving the dust catcher is evidently very much richer in potash than the relatively heavy particles constituting the dust in the dust catchers. However, with the above knowledge at hand, and judging from check calculations on total dust leaving the dust catchers and its theoretical potash content (by difference), it is my opinion that the estimated weight (by difference) of potash lost in the primary washers is somewhat, though not greatly, too high. For our average practice, there should be not less than 12 lb. of total, and probably about 9 lb. of water-soluble potash, now lost in washers and stacks, per ton of pig iron produced.

It may be mentioned that by weak acid treatment part of the insoluble potash content in flue dust may be rendered water-soluble, though this is not likely to be of practical application. Also, the soluble alkali salts can be recovered in tolerably pure form by leaching and evaporation.

It is not my purpose to develop at length the commercial phase of the subject. With the foregoing figures and present potash-fertilizer value as a basis, anyone can readily determine what a very attractive proposition is the recovery of potash from blast-furnace gas at the present time. A word of caution may be appropriate. As previously indicated, the weight of potash

charged per unit of iron produced is above the average at Bethlehem. There are only two or three apparently practical methods for recovery on a large scale of potash from blast-furnace gas, and they are expensive and as yet untried for blast-furnace conditions. The price of potash is certain to fall after the war.

On the other hand, the recovery of potash in connection with the thorough dry cleaning of blast-furnace gas, with certain obvious advantages as against wet cleaning, is an attractive proposition to plants now suffering from burdens rich in alkalies. I venture to predict that in the future dry cleaning will be adopted in many blast-furnace plants, and that many thousands of tons of potash, hitherto wasted, will be reclaimed. Thus will our national resources be strengthened in this important raw material, and the blast furnace will have added another material to its increasing list of by-products.

The subject of alkalies in blast-furnace practice is of a difficult and complicated nature. It will afford much food for thought, both for the philosopher and the practical furnace operator.

Bethlehem Steel Company,  
South Bethlehem, Pa.

## Silica Brick and Some New Uses for Them

By Walter Gray, B. S.

Very little has been generally known about refractories and the adaptability of one kind of material over another for a particular class of work. Engineers have been very well satisfied if the refractory material would reasonably withstand the action of the flame and have not experimented, or investigated with a view of securing the most efficient and economical material. To the advent of the by-product coke oven in the United States is due much of the investigations of refractories which have been made. These experiments and others which have been conducted, indicate quite clearly the advantages of silica brick in many metallurgical and industrial operations. The investigations show that the field for the use of silica is not restricted, and that its adoption may in many cases prove a decided economy.

In order to secure a comprehensive idea of silica as well as its advantages and limitations, it is necessary to compare it with the other available commercial refractories. Such a comparison will show clearly the true worth of silica, and will indicate the probability of its success when substituted for another material. The common commercially available refractories are, magnesite, chrome, bauxite, clay brick, and silica brick.

### MAGNESIA BRICK

Magnesia brick are manufactured from grain magnesite imported from Austria-Hungary which will analyze approximately 85 per cent MgO. The magnesite is quarried, carefully selected, crushed, and calcined at white heat (approximately 2900 deg. Fahr.) in shaft furnaces or rotary calciners. The calcined magnesite upon arrival in this country is tempered, ground with water, and made into brick without bond, and burned in down draft kilns at a temperature approximating cone No. 29. It is thus seen that the ordinary magnesia brick is the result of two burnings.

Magnesia brick have a very high fusing point, but soften and lose their coherence at high temperatures. This objectionable property restricts their use to operations where they will not be subjected to pressure at high temperatures. Magnesia brick expand rapidly and uniformly with an increase in temperature until the maximum is reached at about 2900 or 3000 deg. Fahr.,



where the softening effect is evident. Chemically magnesia brick are basic.

#### CHROME BRICK

Chrome brick are manufactured from chrome ore imported from Greece which will contain approximately 40 per cent  $\text{Cr}_2\text{O}_3$  together with alumina, ferrous oxides, and silica. The ore is ground, formed into brick, and burned in down draft kilns. Chromium oxide is itself not fusible at metallurgical temperatures.

Chrome brick, however, due to the fact that there is but 40 per cent of the oxide present, soften at lower temperatures than magnesia brick and are consequently less strong and less resistant to deformation at high temperatures, and will often if loaded prove unreliable at temperatures above 2800 deg. Fahr. At lower temperatures chrome brick are dense and hard and stand abrasion well, but possess the objectionable feature of spawling with sudden changes of temperature.

Their chief value lies in their chemical neutrality, for chrome brick are neither acid, basic, reducing or oxidizing.

#### BAUXITE BRICK

Bauxite brick are manufactured from the alumina ore of the same name. Pure bauxite is light gray in color, but as it is generally found associated with impurities, the ore is generally red, yellow, or brown. Geologically it is regarded as decomposed basalt, chemically it is a hydrated alumina containing various quantities of aluminium silicate and hydrated oxides of iron.

In order to reduce shrinkage the bauxite has to be thoroughly calcined before being made up into brick and this materially increases the cost. In calcining, the material contracts little, until after the temperature has risen above 2390 deg. Fahr., when excessive shrinkage takes place, the maximum being reached at 2500 deg. Fahr. Water of combination to the amount of approximately 30 per cent of the weight of the raw ore is driven off.

The calcined material is mixed with from one-third to one-sixth part of plastic fire clay and with the addition of water is thoroughly worked and molded into brick and burned at the highest temperatures obtainable in the kiln.

Bauxite brick exhibit a highly basic tendency, the  $\text{Al}_2\text{O}_3$  content running up as high as 55 per cent to 85 per cent. Although the fusing point of bauxite brick is very high, they have the objectionable property of cracking on sudden changes of temperature. By burning at the highest temperatures obtainable in the kilns, much of the shrinkage which was always detrimental to settings or linings made up of bauxite brick, has been done away with, and now bauxite brick are beginning to be used extensively.

#### FIRE CLAY BRICK

We are all more or less familiar with fire clays. Fire clays are the result of decomposition of igneous rocks high in feldspar, which, under the influence of weathering, result in kaolinite. Calcined kaolinite will analyze approximately 46.09  $\text{Al}_2\text{O}_3$  and 53.91  $\text{SiO}_2$ , and is representative of perfect fire clay. These kaolin deposits are stratified upon deposition, and are often subject to considerable pressure, which causes the clay to become hard and dense.

Chemically, fire clays are double silicates of alumina and consist of hydrous silicates of alumina, together with impurities in the form of iron, alkaline silicates, and water, either chemically combined, free, or both. Free silica in excess of the amount required to unite with the alumina will unite with the impurities, act as

a flux, and materially lower the fusing point. The alkaline content should be very low, not over 2 per cent, and the iron should be present in very small quantities, as 5 per cent will make the brick worthless. The amount of iron present is a direct indication of the fusibility of the brick.

The fire clays usually occur underneath the coal measures and require underground mining. The deposits consist of two kinds of clays—the hard flinty clays which are the heat resisting element in the brick, and the soft, plastic clays which form the bond. The hard or block clay, as it is called, very often occurs in the same vein with the soft clay, but is usually above the latter of less depth, and often not separated by any clearly defined line of demarkation. This relationship may reverse itself both as to position and quantity, and sometimes the two are even intermingled. The clays when mined are broken by a hammer, carefully selected, and stocked in piles. The proper proportions of crushed lean and plastic clays, together with "grog," the trade name for ground brick, or calcined clay, are ground in the wet pan with sufficient water to give the required consistency and molded into bricks. When worked between the thumb and finger, it gives a decided sticky feel. The shapes are dried bone dry on a hot floor or in a dryer, and are burned in down-draft kilns at a temperature approximately 2500 deg. Fahr.

#### SILICA BRICK

Before the advent of silica brick, natural siliceous rocks such as sandstones, granites, gneiss, and quartz porphyry had been quarried, carefully dressed, and used as refractory linings. Even at the present time mica schist is still extensively quarried for use in lime kilns. The use of such rocks, however, was not altogether satisfactory; due to physical defects and exfoliation, they suffer severely from the action of the heat. The introduction of silica brick followed quite naturally, for a light uniform shape easily handled and easily laid had everything in its favor, and speedily superseded the natural rock.

#### RAW MATERIAL

Silica brick are manufactured from a sandstone or quartzite which is of sedimentary origin. On account of certain physical features which are very necessary for the manufacture of a well-bonded brick, brick manufacturers are restricted in the selection of their raw material to certain parts of Pennsylvania and Wisconsin and Alabama. For the successful manufacture of brick the siliceous material must be in the form of a rock and not a sand or gravel. The rock should be hard, dense, metamorphic, and semi-crystalline in structure, not granular, with little indications of lime, talc or clay, and with little discoloration from iron. When ground, the grains should appear splintery, sharp, heterogeneous as to form and size, and slightly translucent. It is the physical roughness of the material that gives the brick its strength. The more homogeneously knit together the grains are which compose a brick the better service it will give. Pure quartz, rounded pebbles, or grains of sand, or too coarse crystals prove undesirable as they will not bond tightly. In ganister rock, such as silica brick are made from, the cementing element is as hard as the quartz grains, and upon grinding the particles do not become rounded, but interlock in the brick and bond well.

A suitable quartzite should analyze approximately 97.5  $\text{SiO}_2$ , 1 per cent to 1½ per cent  $\text{Al}_2\text{O}_3$ , 0.75 per cent other impurities, smelt at cone 35 to 36, expand, and swell without perceptible cracking. The total fluxes should not exceed 3 per cent. Alumina, if in the form of kaolin

or a silicate, should probably be not higher than  $1\frac{1}{2}$  to 2 per cent.

#### QUARTZITE

In America we have to distinguish between two kinds of silica refractories: First, brick high in silica, known as ganister or lime-bond silica brick, in which the bonding agent is lime; second, brick lower in silica, known as quartzite, in which clay is the bond used. Although not so strong physically as the clay brick, quartzite possesses the advantage of being easily worked and expands with increases of temperature, but in a much less degree than silica. Quartzite brick will analyze approximately 70 to 75 per cent  $\text{SiO}_2$  and 21 to 26 per cent  $\text{Al}_2\text{O}_3$ . They are manufactured by adding approximately one part of fine ground clay to two parts of crushed quartz, mixing with water and forming into brick.

#### MANUFACTURE OF SILICA BRICK

The two most important deposits of ganister, the trade name for silica rock, best suited for the manufacture of silica brick are found in the Mount Union district of Pennsylvania and the Baraboo region of Wisconsin. The Mount Union rock is fine grained, grayish white in color with tinges of yellow, and showing slight discoloration from iron. The Wisconsin quartzite is much darker in color due to a larger content of iron, and seems much more crystalline in structure. It is denser and harder to work than the Mount Union rock, and has evidently been subjected to a greater pressure, with perhaps some heat. The ganister rock found in the South near Birmingham, Ala., resembles very closely the Mount Union rock, both in its physical and chemical properties. These various rocks will analyze approximately as follows:

$\text{SiO}_2$ .....	0.98%	Lime .....	0.20%
$\text{Al}_2\text{O}_3$ .....	0.60%	MgO .....	0.10%
$\text{Fe}_2\text{O}_3$ .....	0.70%	Alkalies .....	0.40%

Silica rock is almost never mined at depth, but as in the case of the Mount Union district is collected in the form of boulders and fragmentary pieces which have been torn from the parent ledges and strewn over the surrounding territory, sometimes to a depth of 4 ft. or even considerably more, or else it is quarried from the shelves or ledges. When too large these boulders are shattered by blasting and the fragmentary pieces are carefully examined for impurities, and all ferruginous, calcareous or argillaceous rock are rejected. The rock is then transported to large open yards. When required for the manufacture of brick it is crushed to a suitable size in gyratory or jaw crushers. The broken material is then conveyed in buggies to the wet pan, and ground beneath the rollers by a few revolutions of the pan before the milk of lime is added. Only pure soft white lime manufactured from limestones high in calcium carbonate is used. The lime is mixed with the proper quantity of water stored in tubs and fed in proper proportions into the wet pan, usually 2 per cent by weight, and the grinding resumed. Great care is taken to see that the right proportion of lime is added so that it will produce a well-bonded, hard-ringing, clinkered brick. In analysis the lime will often show up as 1.75 per cent, due to losses, but great care is taken that never more than 2 per cent is added, as a material reduction in refractoriness would result.

The material, when ground sufficiently fine, is but slightly damp and, unlike the clays, possesses practically no plasticity, and when worked between the thumb and fingers resembles in feel and appearance moist fine sand. To the trained sense of touch there is a considerable difference in the feel of the material from the different pans, due altogether to the manner in which water has been added and the material worked. The moulder

who receives the ground mixture pounds and works it into the mould with his fingers and trowels it smooth. The moulds are of steel, and make several brick, depending on their size, at the one operation. A steel plate which forms the shelf in a rack car also forms the bottom of the mould, and when the mould is withdrawn it carries the shapes. The rack cars are run to the dryer, or in the case of larger shapes to the hot floor. The drying takes approximately ten to twenty hours, depending, of course, on the size of the shape. Large shapes are laid on the hot floors and turned from time to time. Difficult shapes are less often made in silica than in clay, due in a large measure to the great care required in drying them to prevent cracking, and due also to the lack of plasticity of the material.

The brick are stacked with great care in the kiln, the ordinary standard shapes being placed on edge and bonded at intervals with burned bats. Openings somewhat on the order of checkerwork are left through which the flame can pass and escape through the riddle flues in the floor. The brick are protected from fire flashing by a checkerwork of burned bats built around the firebox. Great care is observed in the setting of the shapes so that they will not only be subjected to the required temperature, but will also sustain the load and not allow a slip to occur, which might prove detrimental to the entire charge. Difficult shapes because of their size or fragility are generally carefully placed high in the kiln where they will have to carry but little weight. The kilns are usually round kilns of the down-draft system, fired with coal, and will hold, depending on their size, anywhere from 30,000 to 100,000 brick at a single firing.

The operation of burning may be divided into three periods. During the first period the brick are thoroughly dried and the moisture removed. This is necessarily a very slow process and cannot well be hastened. Three days are required to drive off the moisture, the temperature gradually being raised to approximately 300 deg. Fahr. Considerable air is admitted throughout this stage of the drying. During the intermediate period, which will last for approximately four days, the temperature is gradually increased and the draft steadily diminished. The carbon dioxide passes off from the lime and the oxidation of any ferrous iron to the ferric condition occurs.

During the burning proper, which forms the third period, the clinker is formed. This clinker is the slag produced by the combination of the lime with the silica. This is a lime silicate, but its character is made more complex by the addition of the  $\text{Al}_2\text{O}_3$  and the  $\text{Fe}_2\text{O}_3$ , which occur in the brick, and unite in forming the slag. This slag glazes over and binds the particles of quartz, and in shrinking makes the brick porous and produces the property of clinkering or producing a ringing sound when struck. During the burning period there is but very little excess air allowed in the kiln, and the temperature is forced up to 2800 deg. or 3000 deg. Fahr. and the brick kept at a soaking heat for three to four days. The final temperature should always be higher than the temperature the brick are to encounter in actual practice. When the brick in the kiln have been kept at the required temperature as indicated by Seger cones for a sufficient time, the dampers are closed, the fires are allowed to die out and the kiln allowed to cool off. Great care is observed to prevent drafts of cold air from striking the heated brick. The kiln necessarily cools very slowly, and ordinarily it is two days after the fires have been extinguished before the kiln can be opened. Four or five days more elapse before the brick are sufficiently cool to handle and sort and wheel to the storage sheds for shipment.



## PROPERTIES OF SILICA BRICK

Very little real scientific investigation has been made with a view of determining the physical properties of firebrick. Many manufacturers have used only a thumb-and-rule method in making their brick, and have secured satisfactory results more by good luck than good management. Indeed, so content have they been with this haphazard system that they have looked upon all experiments in the research laboratory as interesting but not essential, and far too expensive.

Keen competition and the increasing use of refractory materials in many new industrial operations have caused the brick manufacturer in recent years to study his product with a view of determining the adaptability of one refractory over another for a particular class of work. Indeed, the manufacturers are just beginning to realize that the question of refractoriness, vital as it is, is not always the all-important one. Expansion, abrasion, conductivity, and especially the ability of the material to withstand pressure at operating temperatures, must be considered if a highly efficient and economical material is to be produced. It is only within the last few years, with the widespread adoption of the by-product coke oven, that the very important questions of expansion and conductivity have been seriously considered.

La Société d'Encouragement pour l'Industrie Nationale of France, believing that a more comprehensive knowledge of the properties of refractories would be of inestimable value, granted S. Wologdine a research scholarship and placed laboratories at his disposal. With this and one or two other exceptions such experiments as have been made have as a rule been conducted by the brick manufacturers themselves.

## CHEMICAL PROPERTIES

In general, the chemical analysis gives a very good indication of the quality and refractoriness of a brick. The actual fusing point, however, is dependent also in a large measure on the size of the grains, the homogeneity of the mass, the burn, and the mineral elements which comprise the brick. However, in silica, a chemical analysis is of very great importance, as the refractoriness depends directly upon the amount of lime, alkalis and iron present. The silica content should be about 96 per cent, the lime  $1\frac{3}{4}$  per cent to 2 per cent, and the alumina and iron about  $1\frac{1}{2}$  to 2 per cent. As a matter of fact, silica brick will analyze approximately as follows:

SiO <sub>2</sub> .....	96.0%	Lime .....	1.75%
Al <sub>2</sub> O <sub>3</sub> .....	0.90%	MgO .....	0.14%
Fe <sub>2</sub> O <sub>3</sub> .....	0.70%	Alkalies .....	0.39%

Possibly it would be well to explain here the seemingly inconsistent statement that a high-alumina, low-silica firebrick is more refractory than a brick comparatively high in silica, and that a lime-bond silica brick is the equal or superior of a high-alumina fire clay brick. This is explained by the fact that the alumina and silica, which unite to form the alumina silicates of clay brick, are capable of combining in almost any proportion from 1 molecule of alumina to 2 molecules of silica, or from 1 molecule of alumina to 17 molecules of silica (10 alumina to 90 silica by weight). Seger made several tests using cones composed of various proportions of chemically pure precipitated alumina and silica, increasing the silica in multiples of one part of alumina. This relative increase of silica added to one part of alumina was used as ordinates, and the various Seger cones, indicating temperature, were used as abscissæ to plot a curve which would graphically show the fusing point for any combination.

The maximum refractoriness, as indicated by the

highest fusing temperature, is 1 molecule of alumina and 2 molecules of silica which fused at approximately cone No. 37 (3398 deg. Fahr.). The curve drops as we continue adding silica until at about 17 parts silica to 1 of alumina the fusing point is indicated at approximately 3160 deg. Fahr. by cone No. 29. This is the eutectic point of the curve, for as silica is further in-

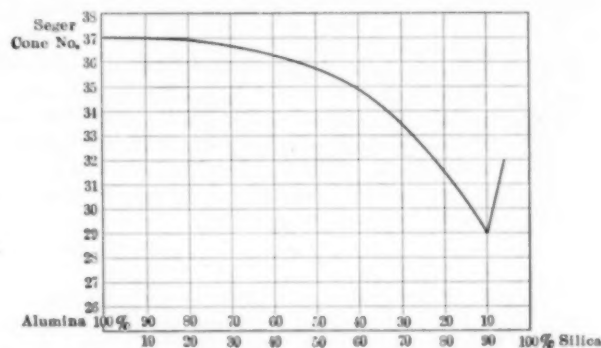


FIG. 1—CURVE SHOWING EFFECT OF ADDITION OF SILICA TO ALUMINA

creased the refractoriness rises until with the proportion of 30 silica and 1 alumina a fusing point of 3300 deg. Fahr. is obtained.

These results are due to the fact that an excess of silica in a clay brick is present in the form of quartz, and acts as an impurity and tends as all impurities do to depress the refractoriness. The curve shows us that we may expect maximum refractoriness in a high alumina brick, such as bauxite, and also in a high silica brick, such as lime-bond silica, and lower refractoriness in a combination of alumina and silica, as in the case of quartzite, which has closely the proportions of alumina and silica referred to as cone No. 17.

Silica, the opposite of magnesia, is acid, and is used in metallurgical furnaces operating with acid slags to form a stable lining. It is very soluble in strong alkaline solutions, easily attacked by metal oxides and flue dust.

## PHYSICAL PROPERTIES

Refractories naturally group themselves into acid, basic and neutral materials, and it is, of course, obvious that where a slag is to be encountered its chemical nature determines to a large extent the refractory material to be used. In other operations, where such limiting conditions do not exist, or at least only in a slight measure, an investigation of the physical properties of the refractories material will assist in determining the best brick to use.

Silica brick are straw colored, smooth of surface, with sharp, well-moulded corners, and present upon breaking a coarse crystalline fracture, discolored occasionally by reddish-brown spots showing the presence of iron oxide. These spots may be due either to iron naturally contained in the rock or may result from the oxidation of small pieces of metallic iron which has been worn from the pan rolls or crusher jaws and been incorporated in the brick.

A 9-in. straight weighs almost exactly 6 lb. and has a specific gravity of 2.4, is quite porous, brittle, and not easily worked.

Silica brick are the ideal brick to use where a high and constant temperature is to be encountered, and where the chemical action of the slag is not detrimental. Silica, however, possesses the objectionable feature of sprawling or disintegrating if subjected to blasts of cold air or irregular cooling when hot. Great care should always be exercised to bring silica brick up to

their working temperature slowly and evenly, for not only does too rapid heating cause excessive spawling, but materially lowers the refractoriness of the brick.

After the original heating, the brick become annealed somewhat, and such great care is not necessary. Indeed, the abuse that silica brick will stand after proper treatment in the original firing is wonderful. Silica brick, like chrome and magnesia, expand when heated, and for this reason are of great value when used in domes or arches or gas-tight chambers, as in expanding they tend to keep everything tight and in alignment. Fire clay brick, on the other hand, contrary to popular belief, expand a slight amount upon heating, but shrink very considerably when cooled, and the fire clay with which they are laid may shrink so much that it opens up the joints and allows the brickwork to settle badly. Silica, even if badly cracked and checked, expands sufficiently to keep the structure tight and in position. Furthermore, silica brick retain their shape and refractoriness practically to the point of fusion, and are capable of supporting loads of 75 lb. per square inch or more at temperatures well above 2700 deg. Fahr. or 2800 deg. Fahr. Magnesia and chrome brick, it will be recalled, soften, and are subject to considerable deformation at high temperatures, while a fire clay brick, if submitted to such a test, would deteriorate and squeeze out of shape.

With standard brick there is no distinction made in regard to grind or burn, as is the case with clay. In shape work the grind is usually finer to insure a more perfect filling of the mould and produce a better appearing shape. The fineness of the grind making a more intimate mixture of the lime and silica resulting in a better slagging effect will increase the strength of the brick. Silica brick are considerable more refractory than clay brick, although not as refractory as chrome or magnesia. They will not melt at temperatures under approximately 3400 deg. Fahr., as compared with 3900 deg. Fahr. for magnesia and 4000 deg. Fahr. for chrome. In silica brick the softening and fusing points are almost identical.

**Expansion.**—Standard 9-in. silica brick will expand as much as  $\frac{1}{8}$  to  $\frac{1}{4}$  in. in length and  $\frac{1}{8}$  to  $\frac{3}{16}$  other dimensions in burning in the kiln, from the green shape to the commercial brick. This increase in volume is accounted for by, first, an increase in the size and number of the pores; second, an expansion of the mineral itself, and, third, the change of the quartz into trydimite. At approximately 1050 deg. Fahr. quartz goes into another crystalline form, then into betaquartz, and at 1800 deg. Fahr. in to trydimite, remaining in this form between 1800 deg. and 2900 deg. Fahr. According to one authority this transformation is accompanied, in the case of a pure quartz, by an increase in volume of approximately 10 per cent. In an ordinary brick, it is believed by many that only one-third to one-half of the whole amount of quartz present has been changed into trydimite, and that the remainder is changed more slowly in the furnace. However this may be, it is always the brick manufacturer's aim to burn all the expansion out of the brick in the kiln, but such a state of equilibrium is never reached. A good silica brick should show a considerable expansion on first burning and a relatively small expansion thereafter.

**Porosity.**—The test for porosity is made by heating the brick to expel all moisture and weighing it, then immersing it in boiling water for several hours, allowing all included air to escape. The difference in weight before and after immersion is a direct indication of the porosity. Silica brick in test will show a porosity of 10 per cent by weight and 20 per cent by volume after immersion for several hours in boiling water.

They are generally more porous than quartzite or clay brick.

**Density.**—The density varies with high temperatures, diminishing as the temperature is increased proportionally to the contraction in the pore space. In the case of silica Wologdine found the density decreased regularly until at 2500 deg. or 2600 deg. Fahr. it was approximately 2.57. This decrease in density is accompanied by only a slight change in porosity. Pure magnesia, on the other hand, shows a marked change in porosity. This change decreases, however, instead of increases, as in the case of silica. The higher the brick is in impurities the lower is the temperature at which the drop in specific gravity becomes manifest. It is evident that a curve correlating burning temperature and density will give a very good expression of the refractory qualities of the brick.

**Permeability.**—As a general proposition the heat conductivity of fire clay and silica refractories is that of the air enclosed in the pores, and not that of the material itself. From this it is evident that conductivity and permeability increase with the porosity and vice versa. It is also evident that the size of the grain and other physical conditions will influence the permeability, the most noticeable of these being the burning temperature. Wologdine discovered that silica brick burned at 1050 deg. C. and had a permeability of 3.3 liters per hour, but at 1300 deg. C. their permeability had increased to 192 liters, and at 1400 deg. C. to 241 liters per hour. This shows that the permeability may vary in a very marked degree, while the other physical properties may change but slightly with the increase of temperature. This is accounted for by the great change in pores and capillary openings due to the transformation of the silica during vitrification.

**Conductivity.**—It is very much to be regretted that the information which is available on this property of refractories is limited to low temperatures. As heat conductivity increases quite rapidly with an increase in temperature, the importance of information on conductivity at operating temperatures is obvious. Experimenters have been limited by the great difficulties of measuring heat at such high temperatures and the physical limitations of the apparatus used. The experiments which have been made have employed temperatures usually not exceeding 400 deg. or 500 deg. Fahr., and only in rare cases have higher temperatures approaching the working condition of the refractory been used. Wologdine reached temperatures of 1000 deg. to 1200 deg. C. in the experiments that he made.

The results of a rather interesting experiment showed that to melt paraffine by heat conducted through  $4\frac{1}{2}$  in. of brickwork 48 minutes were required for clay brick, 45 minutes for quartzite, 39 minutes for silica,  $32\frac{1}{2}$  minutes for chrome and  $17\frac{3}{4}$  minutes for magnesia brick. Another test, using, however, rather low temperatures, showed magnesia to possess the greatest conductivity, with chrome second and silica third, and the clay brick following in sequence in the order of their densities, the lighter densities showing the higher conductivity.

From this, as stated above, we infer that conductivity is a direct function of the density. Density, it will be recalled, is dependent upon the fineness of the grains and the temperatures of burning. It has been shown by experiment that the conductivity of firebrick increases with the temperature of burning; that is, the higher the burning temperature the greater the conductivity. Wologdine found a unique exception to this rule to be chromite, the heat conductivity of this material being independent of temperature. Silica has approximately 20 per cent greater conductivity than clay



brick, whereas the conductivity of magnesia is double that of clay. The increase of conductivity will vary considerably. Wologdine found that bauxite brick increased one and a half times in conductivity when burned at 1300 deg. C., as against 1050 deg. C., whereas in the same range of 250 deg. C. magnesia showed an increase of but 5 per cent.

**Load Test.**—It is generally conceded that the load test is the fairest criterion of refractories. Its chief advantage lies in the fact that it will give a very adequate idea of the behavior of a refractory under operating conditions. Other tests do not always include such considerations. A brick which may show high compressive strength at atmospheric temperatures may fail utterly or possess only a small portion of this strength at high temperatures. A compressive test on cold brick is desirable, but aside from the fact that a weak brick is always a handicap, such a test gives practically no information regarding its resistance to deformation at high temperatures. The load should be somewhat greater than actual furnace loads, as in tests the time element, which is a very vital factor in actual operation, cannot receive attention.

The brick are either loaded singly or built into columns. In either case they are carefully measured in order to calculate the shrinkage and deformation. The results of many tests show that silica is the only brick which will support its own weight, or a reasonable load, without deformation at high temperatures. Quartzite and clay brick begin to show settling at temperatures approximately 400 deg. to 500 deg. Fahr. lower than the temperature at which they were burned. Quartzite brick settle rapidly out of shape from their own weight at temperatures approximating 2700 deg. Fahr. It is evident from this that the range between the softening and fusing points decreases, as the silica contents of a clay brick increase. Silica brick usually show in such tests, especially if built in columns, an increase in height, due to expansion, whereas the clay brick column will show a shrinkage of almost 5 per cent of its original height and the bricks will be fused together. As an illustration, the results of a typical test might be mentioned. Columns were built of silica, quartzite and clay brick, loaded and heated to high temperatures. The silica column supported a weight of 75 lb. per square inch, was subjected to a temperature of 3200 deg. Fahr., and was in good condition at the completion of the test. A similar column made up of clay brick heated to 3100 deg. Fahr. and supporting a load of only 27 lb. per square inch suffered bad deformation. The quartzite column under similar conditions did not prove as reliable as the clay brick.

By plotting a series of failing loads against the chemical impurities in the brick a very good idea can be obtained of the manner in which these impurities affect the refractoriness and resistance to deformation, for it is due to their presence resulting in low fusing points that the test pieces fail.

#### Special Uses of Silica Brick

In calling attention to special uses of silica brick, no claim is made that they will prove a panacea for all the troubles and shortcomings experienced with refractories used in similar metallurgical and industrial practice. Whether or not silica would answer equally well in other operations and under other conditions than the cases to be described is something which only careful study and experiment can determine. Much of the information given was secured from the Harbison Walker Refractories Company. The cases cited are from actual practice and the results described are the observations of operators and men who have been in actual touch

with the plants and conditions under which these silica brick have been used.

In presenting these special uses of silica brick, the only claim that is made is that satisfactory results have been secured from them in the particular uses to which they were put, and that they have in many cases proven an economical substitute. These results, because of their uncommon interest, justify their incorporation in this paper.

#### OPEN-HEARTH CHECKERS

It is interesting to note that the use of silica in open-hearth furnace construction is continually increasing. Formerly the checkerwork was composed entirely of clay brick, and often of not any too good quality. Some steel plants have found it advantageous to use a few courses of silica brick upon the top courses of the checkers. Even the best clay brick when used here fuse and become vitrified. The carbon and particles of slag and limestone carried over by the flue gases permeate and coat them, destroying in a large measure their ability to absorb and give off heat readily.

Silica brick, it is claimed, are less readily choked up, as the carbon coating does not seem to penetrate them and adhere as in the case of clay brick. As they do not fuse or vitrify, being sufficiently refractory to withstand the heat, they not only give many times the life of clay brick, but can be used over several times. Indeed, so favorably have they proven for this class of work that many open-hearth plants are making all their checkerwork of silica, with the exception of the last few courses, which would be subject to spawling due to sudden and wide variations in temperature. Not only is the load on the girder arches considerably less, because of the lighter weight of silica brick, but it is claimed that due to its higher porosity and conductivity the use of silica exerts a marked influence on the quick working of the furnace.

#### BOILER FURNACES

Silica brick have rarely been used in boiler settings and coking arches. Their objectionable property of spawling when struck by a draft of cold air would make them almost prohibitive in such a practice.

They have, however, been tried, and in one or two cases where they were protected from drafts of cold air, and where the heating was gradual and the boilers were not forced, they have given satisfaction and have outlived clay brick, and been quite free from clinkers.

In this connection, it is interesting to note that bauxite brick are being tried in boiler furnaces with gratifying results. They are particularly free from clinkers, such as form on clay brick. Like silica, however, they have a tendency to sprawl on sudden changes of temperature.

#### BEEHIVE COKE OVENS

The use of lime-bond silica brick for the crowns of coke ovens is not new, but possesses some very interesting features. Here we see silica used under conditions which seem most detrimental to its success.

In the original firing some care may be observed in heating the ovens up gradually, but the very nature of coke manufacture makes almost any such attention subsequently impossible. Drawing the oven when at its highest temperature, leaving it to cool very quickly with drafts of cold air impinging on the brickwork, exerts a very detrimental influence. An examination of any coke oven crown will show that the brick have spawled and cracked very badly due to the severe treatment received. However, the expansion of the silica and the taper of the brick retains the pieces in the dome.

When once the crown has become cracked or parted throughout, so that the tendency to spawl is reduced to a minimum, and the brick have become annealed, the crown may be good for a great many years. It seems strange that silica brick used under most trying conditions should be the only satisfactory material for this purpose. Their universal adoption is sufficient proof of their value, as against clay brick, the best of which does not seem able to withstand the high heats for any length of time, but slowly melts and runs, and finally falls from the setting.

#### LIME KILNS

The use of silica brick in the construction of lime kilns is a new departure. On account of its refractoriness and proximity, mica schist has been used extensively for the eyes and piers of lime kilns; indeed, many lime burners say it cannot be excelled. Silica brick, on account of the ease with which they can be moulded into any shape or form and handled, are being tried out by quite a few large lime companies as a substitute for mica schist. The use of silica as a lining material for burning lime may seem paradoxical and contrary to the axioms of chemistry, but when the fact that lime and silica do not combine to form a silicate at the temperatures at which the lime is burned is considered, its use does not seem so unreasonable.

Silica brick have been used generally in the barrel of the kilns, and extend anywhere from a few courses above the eyes to as much as 15 ft. above the arches, the remainder of the stack being clay brick. It is in the barrel of the kiln that the severest action takes place. The hot limestone, when it has worked down this low in the kiln, is semi-plastic and soft and hangs in the shaft concentrating the heat and burning out the lining. The eyes are located at this portion of the kiln and are the weakest point in its construction. They are subjected not only to the intense heat of the fuel and the hot stone, but also to the detrimental effects of drafts of cold air striking the heated brickwork every time the firebox doors are opened.

The kilns of eight large lime companies widely separate, who were probably the first to use silica with any degree of thoroughness, have been selected as representing the most reliable sources of information. Four of these companies manufacture high calcium lime and the other four make a high magnesia lime. Of the high calcium kilns, one is rotary and gas fired, the remainder are of the vertical shaft type and coal fired.

In the rotary kiln silica is used in the hot zone to replace high-grade fire clay and chrome brick, resulting in an increased average life of twelve months, as against a previously maximum of three months. The vertical kilns are lined in the barrel, and in the arches and the piers with silica brick.

With the exception of one kiln, in which the heats were brought up in two hours and no care had been exercised in first firing, the results from using silica brick are most gratifying. One company that is securing approximately twice the life from silica that they had secured from high-grade clay observed great care in firing their kilns. The heats were brought up very gradually for the first twelve hours, and only attained their full intensity after thirty-six hours. After this first firing, however, the silica linings are treated with no more care than ordinary clay brick. In twelve months' service the piers in the kiln had been reduced in cross-section only 25 per cent, due to direct impact of the flame and severe service.

Of the four kilns producing high magnesia lime, but one of them (which is rectangular in shape) is producer-gas fired. It is lined with silica brick in the eyes and

barrel and promises to outlast any previous clay lining. All the other kilns are coal fired. In one kiln, in which silica brick has replaced the arches and jambs, formerly made of mica schist, it gives considerable better life at approximately half the cost, and has proven so satisfactory that this company intends using it in five other kilns.

The largest of these companies, manufacturing high magnesia lime, has one kiln of the Eldred system which was first fired in April, 1911. It is lined in the eyes and barrel with silica brick and has now been in operation for two years and is in such condition that with some minor repairs to the arches, due to sprawling, it may last for a much longer period. The decision of this company to line four more kilns with silica brick is conclusive proof of satisfactory results.

An interesting feature of the use of silica brick at this plant was the failure of the lime to stick or hang, it is claimed, so that considerable of it passed through under-burned, causing a decrease in capacity of approximately 8 per cent a year. This is undoubtedly due to the exceptional refractoriness of silica brick and its failure to in any way flux with the calcining lime. On clay brick the lime will often clinker, forming "monkeys," which in breaking loose tear the brick out with them. By corbelling the brickwork so as to give a purchase for the descending lime this trouble was obliterated, and there has been no recurrence of it either here or at any other plant.

These results indicate that the use of silica brick may mean a decided increase in the life of the kiln lining and result in a considerable economy in operation. Where failures have occurred they can almost always be traced back to injudicious management and poor handling. Where the best results are to be obtained great care must be exercised in the initial heating. The temperature should be brought up gradually and slowly, especially for the first 600 or 800 deg., as it is in the first heating that there is so much danger of spawling. After having been well tempered, silica brick will stand an extraordinary amount of abuse.

#### CUPOLAS

The use of silica brick in cupolas is as interesting as it is unique and uncommon. There are but two or three such cases which have come to the writer's notice.

One company which manufactures steel castings has been using silica brick in its cupolas and for lining its ladles for some four years, and with marked success. This company uses about four or five courses of silica brick in the melting zone of its cupola, and backs them up with standard clay firebrick. The brick are laid tight in silica cement and no extraordinary allowances are made for expansion. In lining its Tronepas converters, however, a space of an inch and a half is allowed against the shell for packing and expansion. No serious trouble has been experienced due to spawling, as the heats are increased slowly. The cupolas are, however, shut down cold each day after a run of ten hours, and in quenching the charge no great pains are taken to prevent the steam from striking the heated brickwork. No accurate records have been kept by this company regarding the life of silica brick, but it is roughly estimated as three to one in favor of silica against high-grade clay, and it is believed this estimate is very conservative.

The ladles used by this company are lined entirely with silica brick, with the exception of the top courses, which are clay brick, as silica would spawl badly at this point, where the hot metal and the atmosphere both react on the brickwork. The linings show a tendency to spawl if heated too suddenly on the first pour. After



a few heats, however, the brick become slightly glazed over, due to the action of the hot metal and the slag, and receive such a hard endurable surface that no further trouble is experienced, no matter how quickly the ladles are heated. Each ladle is used for approximately fifteen heats of 5000 lb. each. The life of silica in these ladles is approximately seven times as great as the life of the clay brick formerly used.

Another company, which on a test secured four weeks' longer life with silica than with any clay brick previously used, have relined their cupola stacks for quite a way up with silica brick. Although the brick have been in but for a short time, they are getting twice the life secured from any clay brick formerly used.

#### BY-PRODUCT COKE OVENS AND GAS RETORTS

Silica has almost entirely replaced quartzite and clay bricks in by-product coke ovens and gas-retort settings. The rapid development of the by-product oven and the thorough investigations and experiments carried on by expert engineers are responsible in a very large measure for the adoption of silica brick. Indeed, the progress which has been made and the results which have been secured are attributed directly by the oven builders to the use of silica.

The physical limitations of clay or quartzite brick do not make their use desirable. In order to secure a high-grade coke, it is important that the chambers should be heated uniformly, and that the coking temperature be high and the time of coking short. It has been found that to decrease the duration of coking has resulted in a much better grade of coke. Also gas of high candle-power is produced in a retort if the charge of coal is large, leaving a minimum amount of retort surface to act upon the gas as it is generated, and if the temperature of the retort is high, resulting in rapid gasification. The hydrocarbons are thus made permanent gases and the tendency to dissociation into other forms of hydrocarbons of lower candle-power minimized. The heavier charges and shorter periods of carbonization have required heats far in excess of that which the old clay retort could stand. Such temperatures as are now employed approach the softening point of clay, and would cause excessive shrinkage and deformation in the old style clay retorts.

The ability of silica brick to stand up and retain their shape at the temperatures encountered in modern gas retorts and by-product coke ovens has made possible the high state of development to which they have been brought. Silica coke oven or retort shapes keep their alignment and neither sag nor bulge. They give considerably longer life than clay shapes and do not have to be renewed so often. As silica has approximately 20 per cent greater conductivity than clay the temperature inside the retort or oven is higher and the time for the distillation of the volatile matter considerably less. The higher the heats, the greater output and the shorter coking time results in a fewer number of benches or ovens for the same plant capacity as compared to clay linings. The expansion of silica brick is a very valuable feature, for if it were not for this property it is doubtful, with sectional retorts and long coking chambers, whether small shapes could be used at all. It is certain, that the expansion tends to keep everything gas tight. In laying the shapes, this expansion is usually taken care of by paper joints. Silica retorts and ovens are not only particularly free from clinkers, but the carbon deposits do not seem to penetrate them as is the case with clay. This results not only in increased heat transference, but saves the brick from severe scarfing action in cleaning, and as the necessity for cleaning is

reduced, the retorts or ovens will be out of commission less frequently.

Actual comparisons between the life of clay and silica in by-product coke ovens are not at hand, but it is a well known fact that silica will give many times the life of clay and in addition prove a great economy in operation. In the case of gas retorts, however, we have a very good comparison. Mr. Samson, in a paper presented before the fourth annual meeting of the American Gas Engineers' Society, described the advantages secured from the installation of silica retorts at the plant of the Worcester Gas Works. The test was made with the same kind of coal which had previously been used in the clay retorts. In the old clay retorts, which were 16 in. x 26 in. x 10 ft., 1 lb. of Westmoreland coal when coked gave 4.95 cu. ft. of 14 cp. gas—practically 70 candle feet. The new silica retorts, which are 26 in. x 26 in. x 17 ft., produced 5.95 cu. ft. of 17 cp.—practically 101 candle feet, from a pound of the same coal. Figures such as these need no further comment; they are themselves conclusive proof of the superiority of silica brick for gas retorts.

#### CONCLUSION

The cases which have been cited involving special or new uses for silica brick are quite representative and cover a wide range of industries using refractory materials. They show quite clearly that the uses of silica brick are by no means restricted. Indeed, it would seem that the advantages and properties of silica are just beginning to be appreciated and that it is only a question of time until silica will replace clay brick in many industries in which it has long been used.

Harbison-Walker Refractories Co.,  
Pittsburgh, Pa.

### Movement for Increased Adoption of Metric System Gaining Headway

During the sixty-ninth meeting of the American Association for the Advancement of Science in New York during Christmas week, 1916, a conference was held, designated the "metric conference," at which were present representatives of national business organizations, the United States Department of Commerce and men prominent in engineering and educational work. The object of this conference was to discuss the Pan-American use of the metric system. Its outcome was the formation of a permanent organization to be known as the American Metric Association, to further the adoption of the international meter, gram and liter, which have been legalized in the United States since 1866.

The officers of the Association are as follows: President, George F. Kunz, 405 Fifth Avenue, New York; first vice-president, William Jay Schieffelin, 170 William Street, New York; second vice-president, E. P. Albrecht, The Bourse, Philadelphia, Pa.; third vice-president, O. E. Stanley, Portland, Ore.; treasurer, Arthur P. Williams, 56 Hudson Street, New York; secretary, Howard Richards, Jr., 156 Fifth Avenue, New York. The executive committee consists of H. V. Arny, 115 W. Sixty-eighth Street, New York; Fred R. Drake, Easton, Pa.; A. E. Kennelly, Massachusetts Institute of Technology, Cambridge, Mass.; S. W. Stratton, Bureau of Standards, Washington, D. C., and W. P. Wilson, Philadelphia Commercial Museum, Philadelphia, Pa.

The association now maintains an office at 156 Fifth Avenue, New York, and is especially desirous of receiving catalogs of manufacturers who use the metric system for domestic or export purposes.

## The Processes of the Organic Chemical Industry Used in the Manufacture of Intermediate Products

### III. Sulfonations and Caustic Fusions

By A. H. Ney and D. J. Van Marle

Sulfonations are the most common of all operations in the organic chemical industry and consist of the action of sulfuric acid on organic compounds, by which hydrogen is replaced by the sulfonic acid group, the products formed being called sulfonic acids.

The result of a sulfonation depends not only on the nature of the product sulfonated, but also on the amount and strength of the acid, the temperature during the sulfonation, and sometimes the time during which the mixture is heated.

The strength of acid used for sulfonating depends on the nature of the product to be sulfonated as there are products, such as benzol, phenol and naphthalene which are sulfonated very easily and therefore can be treated with ordinary concentrated sulfuric acid, and others, such as nitrobenzol, which only can be sulfonated with difficulty, for the sulfonation of which fuming sulfuric acid must be used. During the sulfonation water is formed, which dilutes the sulfuric acid in the mixture, and makes it necessary to use such an excess of acid that its concentration will not fall below the point, where it would become so dilute that it would no longer act on the material to be sulfonated.

The excess of acid required also depends on the temperature during the sulfonation, for, generally, the same results can be obtained with a larger amount of acid or with a stronger acid at a lower temperature as with a smaller amount of acid or a weaker one at a higher temperature. At the same time it must be taken into account that the temperature also has an influence on the position in which the sulfonic acid group enters the molecule and that a higher temperature sometimes causes more than one sulfonic acid group to enter, so that in every case it must be determined with what strength and amount of acid and at what temperature the best results can be obtained most economically. The heat developed is not so great as in nitrations and is mostly caused by the mixing of the acid and the water formed during the reaction. The temperature, at which sulfonations are carried out, is therefore, mostly higher than in nitrations, and cooling generally unnecessary.

In many instances, where different sulfonic acids are formed at different temperatures, the time also has a decided influence on the result of the sulfonation. Phenol, for example, is converted into a mixture of the ortho- and para-sulfonic acids at a low temperature, while at 100 deg. almost exclusively the para-sulfonic acid is formed, and it has been proven that the ortho-sulfonic acid at elevated temperature is transformed into the para-sulfonic acid. This is not an immediate conversion, but is due to the dilution of the acid, this more dilute acid having a reverse action, and splitting off the sulfonic acid group in ortho-position, which then enters the molecule again in the para-position. This phenomenon is especially of frequent occurrence in sulfonations in the naphthalene-series, the result of the sulfonation being an equilibrium between two or more sulfonic acids, the proportion of the different acids depending on the time of heating. Much better results can be obtained here, if the material is not added to the acid, but is fused and heated to the temperature at which the sulfonation has to be carried out, and the acid run in at constant temperature, whereby the equilibrium is reached in a much shorter time.

As to the position in which the sulfonic acid group

enters the molecule, the following rules can be laid down. In the benzene-series the same rule holds true as for nitrations, a sulfonic acid group entering in meta-position to another sulfonic acid group, a nitro-, carboxyl- or carbonyl-group already present. The sulfonic acid in these cases enters with some difficulty and a strong acid has to be used as well as a higher temperature.

If the product to be sulfonated contains chlorine-, alkyl-, hydroxyl- or amido groups, the sulfonic acid group enters into ortho and para position, their proportion depending on the conditions during the sulfonation, especially the temperature and the kind of acid used, the para-compound mostly being formed in much larger quantities than the ortho-compound.

The hydroxyl compounds are sulfonated most easily, resorcline for instance, being immediately converted into a disulfonic acid, the chlorine-compounds not so easily on account of the acid nature of the chlorine group.

The amido-compounds are in an exceptional position, in that they form acid sulfates with the acid, which neutralizes their basic properties, and causes, as in case of aniline and dimethylaniline, the sulfonic acid group to enter both in meta- and para-position. If, however, only sufficient acid is added to form the acid sulfate and this is heated to a temperature of 180-200 deg. the sulfonic acid group is forced exclusively in para position and a uniform product obtained with a minimum amount of acid.

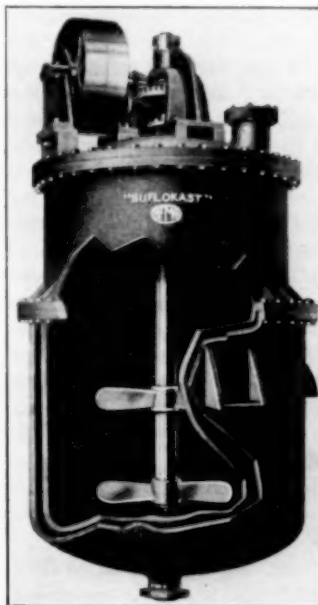


FIG. 1—SULFONATOR

This process, which is known as the baking-process, is used extensively in the manufacture of amido-sulfonic acids.

If two different groups are present before sulfonation, it will depend on their nature, where the sulfonic acid group enters, but if one of these groups is an amido-group its place will be determined by the other group.

In general, ortho-compounds are sulfonated easier than the corresponding para-compounds. Sulfonations in the benzene-series can be mostly carried out in such a way, that a uniform sulfonation product is obtained, which only has to be separated from the excess of sulfuric acid, which can be done by salting out the sulfonic acid or by neutralizing the mixture with lime and filtering off the precipitated gypsum, after which the lime salt is converted into the sodium salt by means of soda ash.

In the naphthalene-series there is a decided difference in the result of the sulfonations, as a mixture of two or more different sulfonic acids is formed. This is caused by the fact that the sulfonic acid group enters first in a position in which it is often not very strongly bound and, therefore, is partly split off again by hydrolysis, then entering in course of the sulfonation into another position.

The temperature and time of heating have a large influence here, at lower temperature, alpha-sulfonic acids, at high temperature beta-sulfonic acids being



formed, their rate of conversion into each other depending on the time.

As there are eight different positions, in which the sulfonic acid group can enter, the situation is very complicated. When naphthalene is sulfonated both the alpha- and beta-sulfonic acid is formed. On further sulfonation the sulfonic acid group enters by preference in the other ring and with hardly any exceptions it never places itself in ortho-, para- or peri-position to another sulfonic acid group.

Alpha nitro-naphthalene is converted into alpha-nitro-naphthalene 5 sulfonic acid, but as the sulfonation is not easy and the same product can be obtained in a different way, this way of preparation has not found any application.

If an amido- or hydroxyl-group is present, the result of the sulfonation will depend on the place of this group, the naphthols being sulfonated very easily, the naphthylamines with more difficulty. They will be discussed in detail later.

To chlorine in alpha-position the sulfonic acid group enters first in position 4, then in 5 and at higher temperature in positions 6 and 7. The manufacture of these chlorosulfonic acids is not of interest, with the possible exception of alpha-chloronaphthalene 4 sulfonic acid, which by caustic fusion can be converted into Neville-Winther acid, which could be done in practice where chlorine is available at a sufficiently low price.

As the result of the sulfonation is a mixture of different sulfonic acids, it will often be necessary in addition to a separation from the excess acid to carry out a separation of the sulfonic acids, which has to be done by means of the difference in solubility of their salts, by salting out these salts or by their difference in behavior toward diazo-compounds.

We will now discuss the influence of the acid and temperature more in detail.

### Sulfuric Acid

Sulfuric acid of the different strengths which are used in the preparation of sulfonic acids, is available in the market. As we have seen, the acid will be diluted by the water formed during the reaction, and the amount for any sulfonation must be such that its concentration, after complete sulfonation, not taking into consideration the amount of sulfonic acid formed, will be high enough to have a sulfonating action on the material.

The sulfonating action of an acid depends in the first place on the degree, to which a product resists sulfonation. All products containing already a group of acid nature, viz., a nitro-, chlorine-, carboxyl- or carbonyl-group, can only be sulfonated with more or less difficulty, and the acid after sulfonation must, therefore, be of a higher concentration, than if the product is sulfonated easily, which is the case with the hydrocarbons and their hydroxyl-compounds.

The alkyl- and amido-compounds are in a medium position.

In the second place the sulfonating action depends on the degree of sulfonation desired, as the sulfonic acid group is of an acid nature and therefore, the molecule resists the entrance of a second and third sulfonic acid group more strongly, so that the concentration of the acid after sulfonation has to be raised accordingly.

It is obvious, that the stronger the acid is, we start with, the smaller will be the required amount, and that it is of advantage for all ordinary sulfonations to use 97-98 per cent sulfuric acid instead of the commercial 66 deg. B. acid, which has a strength of 92-93 per cent.

For instance, for the complete sulfonation of 100 lb. of benzol at 80 deg. C. 415 lb. 92 per cent sulfuric acid have to be taken, whereas, if the 98 per cent acid is

used only 265 lb. are required, in both cases, the concentration after sulfonation falling to 82 per cent, below which benzol is sulfonated only very slowly.

Not only a large amount of acid is saved, but as the excess of acid in the latter case is much smaller, less lime is required for neutralizing the mixture and a smaller bulk of gypsum has to be filtered off, which means in addition a saving in labor.

It would, therefore, be advantageous to use fuming sulfuric acid, of which a still smaller amount would be sufficient, but in the case under consideration this would result in the sulfonation being carried too far, as the first quantities of benzol not only would be mixed with a large excess of highly concentrated acid, but also much heat would be developed by the water liberated on mixing with the strong acid, both of which reasons would cause disulfonic acid to be formed.

To prevent this, it would be necessary to cool the mixture during the first part of the sulfonation, and heating it to the desired temperature after the acid has been sufficiently diluted by the water formed during the reaction, so that the danger of disulfonation has passed.

The foregoing tends to show that there is a lower as well as an upper limit to the concentration of the acid used.

Another way to overcome this difficulty is to start with an acid of ordinary concentration, run in the benzol, and then raise the strength of the acid by adding a sufficient amount of fuming sulfuric acid, as at this stage the dilution will be such as to prevent any disulfonation.

The most economical strength to use will depend in every case on the current price of the available acids. To convert the benzene-sulfonic acid into the disulfonic acid, fuming sulfuric acid containing 20-25 per cent free  $\text{SO}_3$  is required, while at the same time the sulfonation has to be carried out at a higher temperature.

To make the trisulfonic acid a larger amount of this acid has to be used and again it would be of advantage to take a stronger acid, were it not that fuming sulfuric acid, containing 30-55 per cent  $\text{SO}_3$ , is solid at ordinary temperatures and troublesome to handle. Acids with 55-70 per cent free  $\text{SO}_3$  are, however, liquid, and these could be used for reinforcement of the acid, after the sulfonation has been partly completed.

Benzene-trisulfonic acid cannot be sulfonated any further as the sulfonic acid group cannot enter in meta-position to those already present. It is not used for any purpose, as are the naphthalene-trisulfonic acids, which at very high temperature can even be sulfonated to a tetra-sulfonic acid.

Fuming sulfuric acid is also used for those compounds in which the sulfonic acid group enters only with difficulty, such as nitrobenzol, benzoic acid, and amidophenols and for those that have to be sulfonated at low temperatures such as dimethylaniline and many dyes, as the same result can be obtained with a stronger acid at lower temperature as with a weaker acid at higher temperature, providing the temperature has no influence on the nature of the sulfonic acid formed.

In certain cases another point of importance, with regard to the strength of the acid used, has to be taken into consideration, namely, the fact already mentioned, that often the sulfonic acid group enters in a position in which it is not very stable. For instance, in phenol ortho-sulfonic acid, meta-xylol sulfonic acid and meta-cresol sulfonic acid the sulfonic acid group is split off even by boiling with dilute acids, which fact in the last of the three cited cases has found application for the separation of meta and para-cresol. Their mixture is sulfonated, and after diluting the meta-cresol sulfonic acid decomposed by boiling, whereupon the meta-cresol

can be distilled off with steam, the para-cresol sulfonic acid remaining in solution.

This hydrolytic action of the acid also has its influence in the naphthalene-series, where in a number of instances the sulfonic acid group is split off in the position, where it enters first, and afterwards places itself in a more stable position, which is one of the reasons, that in this series, mixtures of different sulfonic acids are formed, the time of heating here having a decided influence on the composition of the mixture. It may, therefore, be necessary to change the strength of acid according to the result desired.

Sulfonations also can be carried out by means of chlorosulfonic acid, which is obtained from sulfur-trioxide and hydrochloric acid gas, or by passing this gas through fuming sulfuric acid and distilling the product. If not used in excess, sulfonic acids are formed and hydrochloric acid liberated, which latter substance, however, introduces serious mechanical difficulties. If used in excess, sulfo-chlorides are formed, the hydroxyl group of the sulfonic acid being replaced by chlorine.

In addition it has the property to direct the sulfonic acid group to a different position than sulfuric acid would and it is in these cases, where a different product can be obtained, that it has found application. Toluol, for instance, by ordinary sulfonation is converted for the greater part into paratoluol sulfonic acid, whereas by the action of chlor-sulfonic acid below 5° C.-60 per cent of the ortho-chlor-sulfonic acid is formed, and which has found a large application in the manufacture of saccharine. In the sulfonation of betanaphthol first the betanaphthol ortho-sulfonic acid is formed which has the sulfonic acid group in position 1, and which under ordinary conditions is immediately transformed into other acids. By dissolving the beta-naphthol in carbon disulfide and submitting it to the action of chlor-sulfonic acid this product, however, can be prepared.

It has been suggested to replace fuming sulfuric acid by a mixture of sulfuric acid and pyrosulfate, but as this introduces a large amount of sodium bisulfate in the sulfonation-mixture, it is not done to any great extent.

There is a class of sulfonations, to which here properly may be called attention, those by means of sodium bisulfite. Amido-compounds form sulfurous ethers with sodium bisulfite, which under certain conditions can be converted into sulfaminic acids and further into amido-sulfonic acids, the sulfonic acid group placing itself in para-position to the amido group, or occasionally in ortho-position. In practice, the starting point for these sulfonations are nitro- or nitroso-compounds, which first are reduced by the bisulfite to hydroxylamine compounds, which also are able to form sulfaminic acids. This reaction has enabled the manufacture of an important intermediate, 1-2 amido naphthol 4 sulfonic acid from beta-naphthol the latter being first converted into nitrosonaphthol.

### Temperature and Time

As already stated, the temperature during the sulfonation is of the utmost importance, not only that the nature of the sulfonic acid depends on it, but also, that it influences the result of the sulfonation in connection with the amount and concentration of the acid. If a product is sulfonated without difficulty, there is danger that more than one sulfonic acid group enters the molecule and the higher the temperature is, the greater this danger, as the action of the acid will be stronger at more elevated temperature, which may make it necessary to cool the mixture, especially in the beginning of the operation, while in many other cases heating is required

to complete the sulfonation. On the other hand, the acid at a higher temperature may decompose part of the product and cause a loss of material. The temperature, therefore, varies according to conditions, ranging all the way from 0 deg. to 200 deg. C.

Since it is most economical to use as concentrated an acid as possible, unless it has an influence on the nature of the sulfonation product the temperature will not be a deciding factor, and will be determined by the strength of acid. In order not to use an excessive amount of acid, the temperature is raised at the end of the operation sufficiently high to complete the sulfonation, time also being saved in this way and the capacity of the apparatus increased correspondingly. In cases where the acid has a corrosive action on the substance sulfonated at higher temperatures, which would impair the quality or the yield of the product, as for example in the sulfonation of beta-naphthol and many dyes, the amount and strength of the acid has to be chosen in accordance with the maximum temperature to which the mixture can be heated.

The heat developed by the reaction itself is not great, so that there is little danger of losing control over the operations as in case of nitrations and reductions, the only result here being that the sulfonation will be carried too far and the material may be partly decomposed.

In the benzene-series, the temperature has little influence on the nature of the product obtained, with the exception of the phenol-sulfonic acids, where the para-compound is almost exclusively formed at higher temperature and in the sulfonation of benzol. Benzene-sulfonic acid is converted up to 90 per cent into the meta-disulfonic acid, the amount of the para-compound which is formed as a by-product, increasing at higher temperature and on longer heating.

It is in the naphthalene-series, that the result of the sulfonation depends entirely on the temperature, the amount and concentration of the acid simply determining the degree of sulfonation.

When naphthalene is sulfonated, a mixture of alpha- and beta-sulfonic acid is formed, and it is not possible to obtain one of these acids exclusively for the reasons mentioned before. At 80 deg. about 80 per cent of the alpha-sulfonic acid is obtained, while at 160 deg. about 85 per cent of the beta-sulfonic acid is formed, the equilibrium gradually changing from one to the other at intermediate temperatures. The old way of preparing the beta-sulfonic acid was to heat the acid to 120-130 deg., add the naphthalene, heat the mixture at 160 deg. for a sufficiently long time to convert the alpha-sulfonic acid into the beta-sulfonic acid and then raise the temperature to 175-180 deg. to drive out the water and complete the sulfonation, which allowed a material saving to be made in the amount of acid required. On the other hand this high temperature caused a small amount of the sulfonic acid to be converted into sultone, while part of the naphthalene escaped sulfonation, as it evaporated with the water.

It is therefore, a great advantage to follow the process, described by Witt, which consists in fusing the naphthalene, heating it to 160 deg. and running in the acid slowly, keeping the temperature throughout at 160 deg. The amount of acid required is larger, as the water is not driven off, but no sultone is formed and no naphthalene escapes sulfonation, so that a higher yield is obtained of a product of better quality.

Another great advantage over the old process is, that the sulfonation is carried out at a uniform temperature of 160 deg. C., so that the maximum amount of beta-sulfonic acid is formed immediately, and no time has to be wasted for converting the alpha-sulfonic acid into the beta-sulfonic acid. It is this feature of the process



which makes it profitable to apply it also in the manufacture of the higher sulfonic acids of naphthalene.

On further sulfonation of alpha-naphthalene-sulfonic acid at lower temperature, the sulfonic acid group enters again in alpha-position, but in the other ring and as position 8 is protected, we will obtain the 1-5 disulfonic acid. In this acid the two remaining alpha-positions are in para-position to those already occupied, so that a third and fourth sulfonic acid group will have to enter in beta-position, of which position 2 and 6 again are protected, being in ortho-position to those taken up. We, therefore, obtain the 1-3-5 naphthalene-trisulfonic acid and 1-3-5-7 tetrasulfonic acid.

If alpha-naphthalene sulfonic acid is sulfonated at a little higher temperature, the sulfonic acid group also will place itself in beta-position, and 1-6 disulfonic acid being obtained, which can further be converted into the 1-3-6 trisulfonic acid and the 1-3-6-8 tetrasulfonic acid.

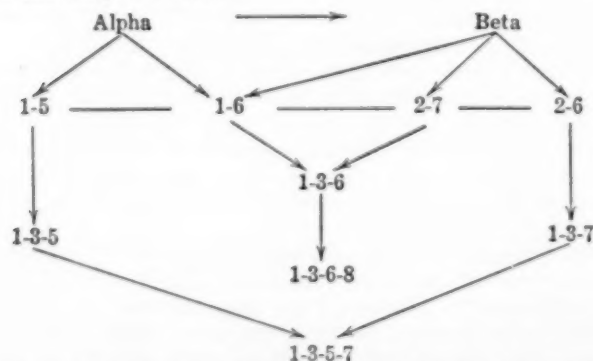
These last two are the only ones that can be formed, and we, therefore, would obtain a uniform sulfonation product if it was not that the 1-6 disulfonic acid will always be contaminated with the 1-5 disulfonic acid, which is formed at the same time and with the disulfonic acid, obtained from the small amount of beta-sulfonic acid, present in the alpha-acid.

To purify the 1-6 disulfonic acid first would be tedious and it is generally preferred to postpone any purification until the ultimate product has been prepared.

In the beta-sulfonic acid the sulfonic acid at lower temperature can enter into alpha-position, which will give us the 1-6 disulfonic acid, at higher temperature into the beta position, when we will get a mixture of the 2-6 and 2-7 disulfonic acids.

All these disulfonic acids are convertible into each other, the 2-6 acid being the most stable at the highest temperature. Both the 1-6 and 2-7 disulfonic acids can be converted into the 1-3-6 trisulfonic acid and the 1-3-6-8 tetrasulfonic acid, while the 2-6 disulfonic acid forms the 1-3-7 trisulfonic acid, which like the 1-3-5 trisulfonic acid goes over into the 1-3-5-7 tetrasulfonic acid. In case of the 2-6 and 2-7 disulfonic acids, the two remaining beta-positions are protected and another sulfonic acid group, therefore, has to enter in alpha-position, even at higher temperature. In the 1-3-6-8 tetrasulfonic acid we have an exception to the rules, in that the sulfonic acid group places itself in position 8, or in peri-position to another sulfonic acid group.

The preceding can be readily followed with the aid of the diagram below:



As pointed out Witt's process here also will give more favorable results, as, for instance, the beta-sulfonic acid can first be prepared, and converted at 160 deg. into the disulfonic acid by an additional amount of acid, the maximum amount of 2-7 disulfonic acid then being obtained, which subsequently can be converted into the 1-3-6 trisulfonic acid by reinforcing the acid.

The naphthols are sulfonated very easily, and care has to be taken that the temperature is not carried above

the point where the corrosive action of the acid will become noticeable.

Sulfonation of alpha-naphthol yields a mixture of the 2 and 4 sulfonic acids, and the 2-4 disulfonic acid, while at a little higher temperature the 2-4-7 trisulfonic acid is formed with a small amount of 2-4-6 trisulfonic acid. The sulfonic acid groups in positions 2 and 4 are not very strongly bound, which causes the 2-7 and 4-7 disulfonic acids also to appear in the sulfonation-mixture. If the hydroxyl-group is protected, however, which can be done by converting the alpha-naphthol into alpha-naphthol carbonate the sulfonation will go much more smoothly, first the 4 sulfonic acid being obtained, then a mixture of the 4-6 and 4-7 disulfonic acids, in analogy to the sulfonation of alpha-naphthylamine. It appears that the sulfonic acid group in alpha-naphthol derivatives never enters in position 3, 5 or 8.

In beta-naphthol the sulfonic acid group first enters in position 1, in which it is so unstable that it is immediately converted into the 8 sulfonic acid or Croceine acid which in its turn at higher temperature goes over into the 6 sulfonic acid or Schaeffer acid. On further sulfonation these two acids form the 6-8 disulfonic acid or G-acid, and the 3-6 disulfonic acid or R-acid respectively, both of which are generally prepared by direct sulfonation of beta-naphthol at different temperatures and both of which ultimately go over into the 3-6-8 trisulfonic acid. The beta-naphthol 7 sulfonic acid can be sulfonated to a mixture of the 1-7 and 3-7 disulfonic acids, which can be converted into the 1-3-7 trisulfonic acid and ultimately into the 1-3-6-7 tetrasulfonic acid, which is the only case in which the sulfonic acid group enters into orthoposition to another one.

Alpha-naphthylamine is converted into a mixture of the 4 and 5 sulfonic acids, the latter being obtained in greater amount with fuming sulfuric acid at a low temperature, and the former most at higher temperature, while at the same time a small amount of the 6 sulfonic acid is formed. The 4 sulfonic acid or naphthionic acid, which is the most important, is made preferably according to the baking-process. By further sulfonation it goes over into a mixture of the 4-6 and 4-7 disulfonic acids, generally known as Dahl's acids II and III, the 5 sulfonic acid into a mixture of the 2-5 and 5-7 sulfonic acids, both of which can be converted into the 2-5-7 trisulfonic acid.

In the other alpha-naphthylamine sulfonic acids, which can mostly be obtained by nitration and reduction of the naphthalene-sulfonic acids, a second sulfonic acid group enters in position 4 or 5, a third one in position 2 or 7, as can be seen from the following table:

Sulf. Acid	Disulf. Acid	Trisulf. Acid
2	2-5	2-5-7
3	3-5	3-5-7
6	4-6	2-4-6
7	4-7	2-4-7
8	4-8	2-4-8

The alpha-naphthylamine 5-7 disulfonic acid, which is of technical importance can be prepared by sulfonation of alpha-acet-naphthalide, the acetyl-group protecting position 2 and 4, which causes the sulfonic acid group to enter exclusively in position 5, the 5 sulfonic acid then being converted into the 5-7 disulfonic acid only.

The sulfonation of beta-naphthylamine is very complicated, a mixture of the 5, 6, 7 and 8 sulfonic acids being formed, the 5 and 8 sulfonic acid to a greater degree at lower temperature, being alpha-sulphonic acids, the others at higher temperature. It is hardly possi-

ble to prepare them in a pure state, except the 5 sulfonic acid, which can be obtained by sulfonation of beta-acetnaphthalide. The others are preferably made by replacing the hydroxyl-group by the amido-group in beta-naphthol sulfonic acids by means of ammonia. A second sulfonic acid group enters often in position, in which position, however, it is not stable, and may also enter in the same ring in meta position to the first one.

### Caustic Fusions

Closely connected with the sulfonations are the caustic fusions, which convert the sulfonic acids into hydroxyl-compounds by fusing them with caustic soda. The sulfonic acid, probably under influence of the caustic soda, first goes over into an ester of sulfurous acid, which subsequently is decomposed into a phenolic body and sodium sulfite. This reaction takes place only at a high temperature, which makes it necessary to use an excess of caustic soda to keep the mixture in a liquid state at this temperature, but as many organic compounds are easily oxidized in the presence of caustic soda at a high temperature, especially if there are any amido-groups present in the molecule, a considerable amount of material is lost through oxidation and the yield leaves much to be desired. This can often be improved by carrying out the caustic fusion with water

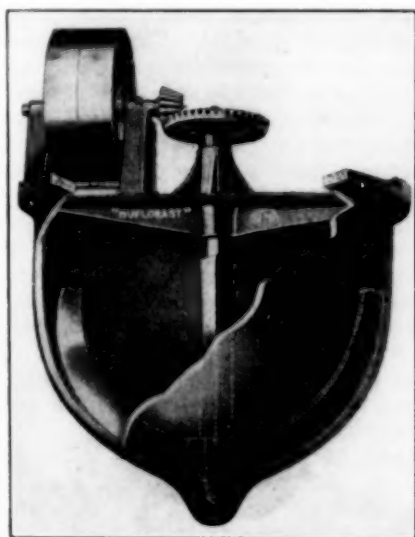


FIG. 2—FUSION KETTLE

in autoclaves under pressure, which allows the amount of caustic soda to be cut down materially and prevents oxidation. The disadvantages of using autoclaves is their limited capacity and the long time required for their operation, wherefore they are used only in such cases where the materials are more expensive or the amount of caustic soda required for fusion in open kettles is excessive.

In the manufacture of carbolic acid from benzene-sulfonic acid and beta-naphthol from beta-naphthalene sulfonic acid, which are both manufactured in very large quantities, the use of autoclaves, therefore, is not practicable. These two products, being mono-sulfonic acids the excess of caustic soda required is not large, it having also been found in the case of carbolic acid that pressure-fusion has no favorable influence on the yield. In the case of di- and trisulfonic acids, however, a much larger excess of caustic soda has to be used, and here generally the use of autoclaves will be preferred. These products at the same time are the more expensive ones, and mostly not prepared in such large amounts. In

these higher sulfonic acids the sulfonic acid groups are not all at once replaced, benzene meta-disulfonic acid, for instance, first being converted into phenol meta-sulfonic acid, then into meta-dioxybenzol or resorcin. A peculiarity of the caustic fusions is that sometimes the hydroxyl-group not always enters in the same position as the sulfonic acid, which it replaces, which can be illustrated by the fact that benzene-ortho- and para-disulfonic acid by fusion with caustic soda also are converted into resorcin.

In the naphthalene-series the sulfonic acid groups in alpha-position are, as a rule, easier replaced by the hydroxyl-group than those in beta-position, which has made the manufacture of amido-naphthol sulfonic acids from naphthylamine sulfonic acids possible. Especially those of 1-8 amido-naphthol such as H-acid and 2-8 amido-naphthol, such as Gamma acid, are of great practical importance. The replacement of a sulfonic acid group in alpha-position is especially easy, if it is in position 8 to an amido-group or in meta-position to another sulfonic acid group. If these fusions of naphthylamine-sulfonic acid are carried out in the presence of water it must be taken into consideration that the amido-group also may be replaced by the hydroxyl-group, due to the hydrolytic action of the water, which may cause the 1-8 amido-naphthol 3-6 disulfonic acid or H-acid, for example, to be converted into 1-8 dioxy-naphthalene 3-6 disulfonic acid or chromotropic acid. A few of the naphthalene sulfonic acids also are converted into naphthol-sulfonic acids by caustic fusion.

Of the fact, that the material can be oxidized in the presence of caustic soda advantage has been taken in the manufacture of alizarine and other anthracene dyes. Alizarine is made by sulfonation of anthra-quinone and caustic fusion of the sulfonic acid, the mono-sulfonic acid hereby being converted into dioxy-anthraquinone or alizarine. Usually the oxygen needed for the oxidation is not supplied by the air alone, but partly by an oxidizing agent, such as sodium chlorate or sodium nitrate.

Not only the sulfonic acid group but also the chlorine-group under certain conditions can be replaced by the hydroxyl-group. If this could be done in chlorbenzol we would have a very simple process for the manufacture of carbolic acid, but the temperature at which the chlorine-group here is replaced is so high that the resulting pressure is too high to make the process of commercial interest. If any acid groups are present in the molecule, however, the conversion becomes easier and can be carried out under workable conditions. So, for instance, in ortho- and para-nitro-chlorbenzol, the chlorine is made removable by the presence of the nitro-group, making it possible to convert them into ortho- and para-nitrophenol. If two nitro-groups are present, as in dinitrochlorbenzol, the conversion is so easy that boiling with caustic soda is already sufficient to form dinitrophenol. In the naphthalene-series we find an example in alpha-chloronaphthalene 4 sulfonic acid, from which alpha-naphthol 4 sulfonic acid, an important intermediate, may be prepared.

That the sulfonic acid group is not always replaced, we can see in the case of naphthionic acid, which by the action of concentrated caustic soda solution under pressure, can be converted into alpha-naphthol 4 sulfonic acid, the amido-group being replaced instead of the sulfonic acid group. The conversion is not easy, however, and the process has been practically abandoned.

To obtain the hydroxyl-compounds in a pure state, the fusion mixture is dissolved in water and the solution acidulated, the acidulation being carried only so



far that the sulfite is converted into bisulfite. If the hydroxyl-compound is soluble, as is often the case, it can then be precipitated by addition of salt to the solution. Resorcline forms an exception, in that it is soluble in water and cannot be salted out. To obtain it in a pure form, the solution is extracted with an organic solvent and the crude resorcline distilled in vacuum.

### Application

The sulfonic acids of benzol are only manufactured as intermediate products in the production of phenol and resorcline, into which the mono- and disulfonic acid respectively are converted by caustic fusion. The first is only used to a small extent in the preparation of dyes, but in enormous quantities as an antiseptic and in the manufacture of artificial resins and picric acid. Its production during the year 1916 was estimated at 10,000 tons. Nitroso-phenol, which enters in the manufacture of a few dyes, can be made from phenol by the action of sodium nitrite. Salicylic acid, which also can be made from phenol, has found application both in the dyestuff industry and for medicinal purposes, as has resorcline. To make picric acid from phenol it is necessary to sulfonate the phenol first, and it is possible to make it from either the mono-sulfonic acid or the trisulfonic acid. The first is treated with a large excess of nitric acid and does not give a very good yield, while the second, which has to be made with fuming sulfuric acid and, therefore, is more expensive, can be converted into picric acid with only a slight excess of nitric acid or its equivalent in sodium nitrate and gives a better yield.

Ortho-toluol sulfochloride, which is formed by the action of chlorsulfonic acid on toluol, is the starting point in the manufacture of saccharine. The para-compound is obtained as a by-product and has found application in the form of its methyl- or ethyl-ester as an excellent alkylating agent.

The sulfonic acid of nitrobenzol is converted by reduction into metanilic acid, which is mainly used in the manufacture of metanil yellow. The metanilic acid by caustic fusion gives meta-amidophenol, one of the intermediate products in the preparation of the Rhodamine dyes. The alkyl compounds of meta-amido phenol, which also are used for this purpose, are obtained by sulfonation of the alkyl-compounds of aniline with fuming sulfuric acid at low temperature, followed by caustic fusion.

Para-nitrotoluol sulfonic acid is the basic material for the manufacture of the stilbene-dyes, chrysophenine and brilliant-yellow. Of other nitro-sulfonic acids those of nitrochlorbenzol deserve attention. The chlorine-group here is unstable and can be replaced by the hydroxyl-group by means of caustic soda. On reduction, we then can obtain the amidophenol sulfonic acids, the ortho-amidophenol sulfonic acid giving important chrome-dyes of excellent fastness. In nitrochlorbenzol itself, the chlorine-group can also be replaced by the hydroxyl-group, as we have seen. Of the nitrophenols formed, the para compound can be used for manufacturing para-amidophenol, the ortho compound for anisidine and dianisidine. The nitrochlorbenzol-sulfonic acids can also be converted into nitraniline-sulfonic acids, from which a few lake-dyes are derived.

The sulfonic acids of amido-compounds, such as aniline, toluidine, xylydine, amidoazobenzol, meta-phenylene diamine and meta-tolylene diamine are intermediate products for the manufacture of many azo dyes. Ethyl-benzylaniline sulfonic acid is more used for triphenylmethane-dyes.

Naphthalene-sulfonic acids are only intermediate products in the manufacture of other naphthalene-compounds. By caustic fusion they can be converted

into naphthols and naphthol sulfonic acids. The most important one is the beta-naphthalene sulfonic acid, the first stage in the preparation of beta-naphthol, which is used in enormous quantities for making beta-naphthylamine, beta-naphthol sulfonic acids, para red and many other dyes. Alpha-naphthol generally is not made from alpha-naphthalene sulfonic acid, but from alpha-naphthylamine sulfate by heating this with water under pressure. Its application is limited, as we have seen that on sulfonation an inseparable mixture of sulfonic acids is formed. This mixture on nitration can be converted into naphthol yellow, the most important dye made from alpha-naphthol. Other naphthol-sulfonic acids, obtained from naphthalene-sulfonic acids, are Cleve's alpha-naphthol 5 sulfonic acid, alpha-naphthol 3-6 sulfonic acid GR and beta-naphthol 7 sulfonic acid F. The first two are used for azo-dyes, the last one for preparing a blue shade of para-red and for making beta-naphthylamine 7 sulfonic acid.

By nitration, followed by reduction, the naphthalene-sulfonic acids can be converted into alpha-naphthylamine sulfonic acids. The most important are alpha-naphthylamine 8 sulfonic acid from the alpha-sulfonic acid, the 5 sulfonic acid or Laurent acid being obtained as a by-product; a mixture of Cleve's alpha-naphthylamine 6 and 7 sulfonic acid from the beta-sulfonic acid and Freund's alpha-naphthylamine 3-6 disulfonic acid from the 2-7 disulfonic acid, all of which are used in the manufacture of azo-dyes. Others are not used as such, but only for the preparation of other intermediate products. The amido-group, for instance, can by different means be replaced by the hydroxyl-group, thereby forming alpha-naphthol sulfonic acids, which is done for the preparation of alpha-naphthol 3-8, and 4-8 disulfonic acids and the 3-6-8 trisulfonic acid.

Of the alpha-naphthylamine sulfonic acids, obtainable by direct sulfonation, naphthionic acid is the most important, and used in large quantities for preparing azo-dyes and in the manufacture of Neville Winther's alpha-naphthol 4 sulfonic acid. Laurent's alpha-naphthylamine 5 sulfonic acid is not made by direct sulfonation, as it then is not pure, and it is obtained in sufficient quantities as a by-product in the manufacture of the 8 sulfonic acid.

The beta-naphthylamine sulfonic acids, as already mentioned, can only be prepared in a pure state from the beta-naphthol sulfonic acids by means of ammonia. All naphthylamine di and trisulfonic acids can be converted into amidonaphthol sulfonic acids by caustic fusion. The most important is H acid or 1-8 amidonaphthol 3-6 sulfonic acid, obtained by nitration and reduction of naphthalene 1-3-6 trisulfonic acid followed by caustic fusion. Gamma acid or 2-8 amido-naphthol 6 sulfonic acid is next in importance and can be prepared by converting beta-naphthol 6-8 disulfonic acid G into the corresponding beta-naphthylamine compound and fusing this with caustic soda. Others are 1-8 amido-naphthol 4 sulfonic acid S, 2-4 disulfonic acid S, and 4-6 disulfonic acid K, 1-5 amido-naphthol 7 sulfonic acid M, 2-7 amido-naphthol 7 sulfonic acid J, and 2-8 amido-naphthol 3-6 disulfonic acid RR, which all are made from the corresponding naphthylamine sulfonic acids. The 1-2 amido-naphthol 4 sulfonic acid is prepared from nitroso beta-naphthol with sodium sulfite, and the amido and hydroxyl group being in ortho position to each other, it gives valuable chrome dyes.

The beta-naphthol sulfonic acids, which have been mentioned, also are of great importance. We obtain a mixture of the Croceine and Schaeffer acid or of the R and G acid, of which the Croceine and G acid and their salts are much more soluble than the other two, of which property we can make use for their separation, in that the salts of Schaeffer and R acid can be pre-

precipitated by adding salt to their solutions. The precipitate can be washed with salt-solution free of the other component acid which separation is quite satisfactory for preparing Schaeffer and R salt. The precipitation, however, is not complete and the other two acids can, therefore, not be purified in this way. As they both contain a sulfonic acid group in position 8, they combine much slower with certain diazo-compounds, especially diazo-xylol, than Schaeffer and R salt. Their purification is carried out by combining these two acids with diazo-xylol or another suitable diazo-compound and salting out the dye found, the pure Croceine or G salt remaining in the mother-liquor, from which they can be obtained by evaporation.

Further sulfonic acids of the naphthalene-series are 1-8 dioxy-naphthalene 3-6 disulfonic acid or chromotropic acid, obtained by replacing two sulfonic acid groups in 1-3-6-8 naphthalene tetra sulfonic acid with caustic soda; or by fusing alpha-naphthol 3-6-8 trisulfonic acid with caustic soda; 1-8 dioxy-naphthalene 4 sulfonic acid, made by replacing the amido by the hydroxyl-group in 1-8 amido-naphthol 4 sulfonic acid, and the sulfonic acids of dioxy-naphthoic acid. These are prepared by converting alpha- or beta-naphthol into the oxy-naphthoic acid in the same way as salicylic acid is made from phenol, sulfonating this product and fusing the disulfonic acid with caustic soda.

All these sulfonic acids of the naphthalene-series are almost exclusively used in the manufacture of azo-dyes.

Sulfonations and caustic fusions are also carried out to a large extent in the manufacture of many anthracene-dyes, the simplest of which is alizarine, made by sulfonation of anthraquinone and caustic fusion of the sulfonic acid in the presence of an oxydizing agent, whereby dioxyanthraquinone is formed, which is alizarine, the well-known substitute for Madder.

Dyes are often sulfonated to increase their solubility or improve their quality otherwise. The best known examples are alkali blue, soluble blue, water blue, patent blue, primuline, induline and nigrosine.

### Apparatus

Sulfonations are carried out in kettles of acid-proof cast iron, provided with an agitator. With the exception of benzol and toluol, all materials to be sulfonated in the ordinary way are solids and as the sulfonic acids are all soluble in the excess acid the sulfonation mixture is homogeneous, so that a simple agitator of the propeller or horseshoe type will be sufficient. In the case of benzol, however, the agitation has to be very thorough, as benzol is immiscible with and very much lighter than sulfuric acid. The best type of agitator is a high speed propeller placed inside a cylinder, which is perforated at the bottom. The propeller is run in such a direction that it takes the benzol down from the surface, mixes it with the acid and forces the mixture through the openings at the bottom and causes it to rise along the sides of the kettle. In general, sulfonations are very simple, and can be carried out without difficulty, the acid being run into the kettle, the material being added gradually and the mixture being kept at the desired temperature until the sulfonation has been completed.

As we have seen, sometimes the mixture is cooled during the first part of the operation and heated afterward, generally by means of steam, which can be easily done by providing the sulfonating kettle with a jacket. If steam-pressure is not sufficient to reach the desired temperature, the heating can be done by means of an oil-bath, which is heated directly. Wherever a battery of kettles has to be heated, it is more economical to circulate oil through the jackets of the kettles and have a

central heating system. If a sulfonation at high temperature is followed by a nitration, the sulfonating kettle has to be oil-heated and the mixture has to be transferred to a second kettle, in which it can be cooled and nitrated. It would be of advantage to carry the operation through in one kettle, which can be done if the kettle has a heating-coil cast in, through which first pressure-water is circulated which, after the sulfonation is complete, is gradually replaced by cold water.

The sulfonation of amido-compounds is often carried out, as already mentioned, by heating the acid sulfate at a temperature of about 200 deg., which is done in an oven in which this acid sulfate is placed in trays on shelves, which are heated by means of furnace-gases. The operation can be considerably facilitated by conducting the operation in a vacuum.

The separation of the excess sulfuric acid from the sulfonic acid can be done in three different ways. If the sulfonic acid is not soluble in dilute acid, as in case of beta-naphthalene sulfonic acid the sulfonation mixture can be diluted with water, which causes the sulfonic acid to precipitate. If a large excess of sulfuric acid is used, the sulfonic acid can often be precipitated by adding salt to the diluted sulfonation mixture, which sometimes effects a separation of sulf. acids of different solubility at the same time. In both cases large amounts of dilute acid have to be handled and acid-proof filtering devices, which are yet far from perfect, have to be used. If the sulfonic acid, and its salts, however, are too soluble under these conditions, or if it is preferred not to handle any acid liquors, the sulfonation mixture can be neutralized with lime, the sulfuric acid being precipitated as gypsum, which can be filtered off in ordinary filterpresses and washed. The sulfonic acid then is obtained in form of its lime salt, which is converted into the sodium salt by means of soda ash, as the lime salts generally cannot be used as such. If a caustic fusion follows the sulfonation, the lime would cause the fusion-mixture to become very thick, and a large excess of caustic soda would be required. Many sulfonic acids are used directly in the manufacture of dyes, especially azo-dyes, and here the lime might often cause the dye to precipitate as a lime salt, insoluble in water.

The sodium salts of the sulfonic acids are soluble in water, which solution sometimes can be used immediately in the manufacture of dyes. If a mixture of sulfonic acids is formed, it may be desirable to separate them, before they are used further, which can often be effected by salting out the sodium salt or the acid sodium salt of the least soluble sulfonic acid. For all

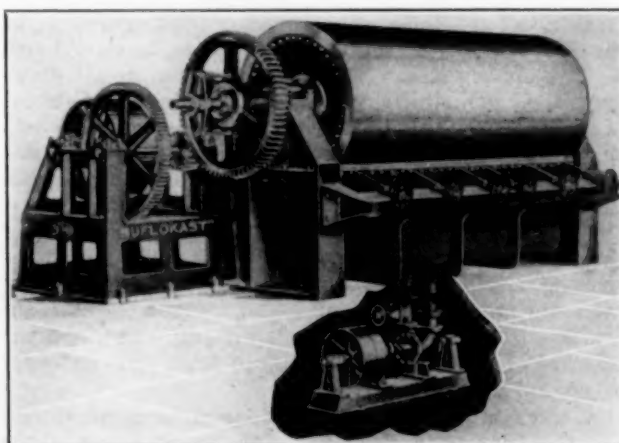


FIG. 3—ATMOSPHERIC DRUM DRYER



these operations, following a sulfonation, only wooden or lead-lined tanks and filtering apparatus are required.

In the manufacture of carbolic acid the sodium sulfonate of benzol is obtained in solution, which must be evaporated to dryness. As enormous amounts of this product are produced at the same time, it is important to do this in the most economical way, which consists in concentrating the solution in a vacuum-evaporator and evaporating the concentrated solution to dryness in one operation on an atmospheric drum dryer, thereby bringing down the cost of this operation to a minimum. This apparatus can be used to advantage in all similar cases wherever the sodium salt of a sulfonic acid has to be prepared in the dry state.

Caustic fusions are carried out either in open cast iron kettles, in which the caustic soda is fused and the sodium sulfonate gradually added or in autoclaves under pressure. For pressures up to 500 lb. these autoclaves can be made of cast iron, but for higher pressures they have to be made of cast steel. Both autoclaves and fusion kettles are provided with an agitator and are placed in a furnace, which is heated by oil or gas, as the temperature to which the mixture is heated is often above 300 deg. C. When the fusion is finished the mixture is dissolved in water. The contents of an autoclave are sufficiently liquid to blow them into water by air-pressure, but in case open kettles are used the mixture is generally ladled out, allowed to solidify and then dissolved. It has been tried to provide these fusion kettles with a bottom-outlet, in order to run out the mixture, but as it is difficult to keep these bottom openings tightly closed and as they burn out gradually, they have not given much satisfaction. The operations following a caustic fusion, consisting in acidulating the solution of the fusion mixture, separating out the hydroxyl-compound if necessary, and filtering this off, requires only wooden tanks and simple filtering devices. Carbolic acid separates out as a liquid, and the acidulation is, therefore, carried out in iron tanks with conical bottom. The soluble hydroxyl compounds can be purified by recrystallization. Phenol, alpha- and beta-naphthol are purified by distillation, the latter two in vacuum.

50 Church St., New York City.

## Synopsis of Recent Metallurgical and Chemical Literature

### Refrigeration

**The Steam Jet Refrigerating Machine.**—In the *Electric Journal* for January, J. C. BERTSCH of the Westinghouse Machine Co., describes the Leblanc refrigerating machine which produces refrigeration by the evaporation of water. The process is an old one, strictly speaking, but its successful commercial development is comparatively recent. To Maurice Leblanc, of France, is given the credit for perfecting this machine, and developing the high vacuum-producing and vapor displacing devices known as the Westinghouse-Leblanc ejector and air pump. With the use of these appliances, the refrigerating process consists simply of the evaporation of a part of the water contained in the substance to be cooled or concentrated under a very high vacuum, which is produced by steam jets; the condensation of the vapor and working steam and the removal of the air and condensate from the condenser by special pumps.

A diagram of the apparatus as used for general purposes is shown in Fig. 1. For temperatures above 35 deg. F., as required for air conditioning plants, drinking water systems, rubber rolls, etc., water alone

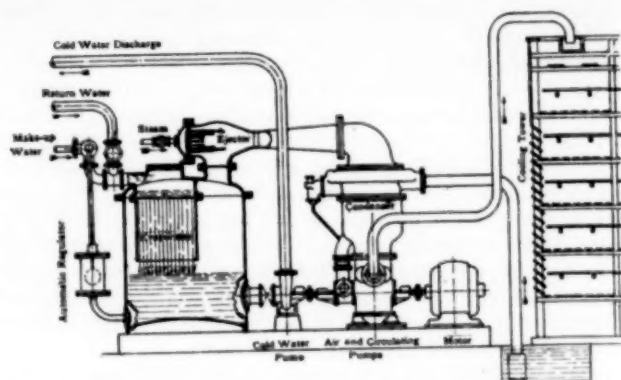


FIG. 1—TYPICAL ARRANGEMENT OF LEBLANC REFRIGERATING MACHINE

is used as the cold carrier, but for temperatures below 35 deg. F., salt brine is used as carrier.

The evaporator is divided by a cylindrical partition into a brine and vapor space, the former has near the top a perforated horizontal partition as shown, through which the warm brine from the cooling coils is returned to the evaporator in the form of a spray. The make up regulator, consisting of a float valve, controls automatically the amount of makeup water needed to replenish the water lost by evaporation.

The evaporation under vacuum and subsequent cooling is produced by the steam ejector shown at the top of the vapor space. This is the most important part of the apparatus, as it maintains a high vacuum and removes the great quantities of vapor necessary. It is made up of a large number of small De Laval steam nozzles, which give a high velocity. The condenser is a steam condenser as commonly used in connection with turbines and steam engines. A cooling tower is very desirable. A specially designed Leblanc air pump is used for removing the air in the condensed water. For the circulation of the cold brine a cold water pump (centrifugal type) is used as shown. The entire absence of chemicals, the automatic operation, and the limitation of the moving parts to a few centrifugal pumps, make very little attention necessary. Actual tests on a 60-ton machine in a rubber plant showed a cost per ton of refrigeration of \$0.304 to \$0.334, producing 72-79 tons per day. Besides chemical and sugar works, the machine is specially suited for marine works.

### Iron and Steel

**Grain-growth in Low Carbon Steel.**—At the eighty-first meeting of the Faraday Society on Dec. 18, 1916, in London, a paper on "Grain-growth in Deformed and Annealed Low Carbon Steel," presented by RALPH H. SHERRY of Detroit, U. S. A., was read by Sir Robert Hadfield for the author.

Coarse crystallization or grain-growth in pure iron and low-carbon steels permanently deformed and annealed has from time to time caused no little difficulty to workers in sheet, wire, cold-drawn bar and pressings of these materials. The present paper, based on an extended investigation, explains the conditions under which grain-growth occurs.

The author summarizes his results as follows:

1. Grain-growth in low-carbon steel will be produced by permanent deformation of the metal within certain limits, followed by annealing within certain temperature ranges.
2. The greater the amount of strain within these deformation limits, the smaller will be the grain-size produced by annealing.
3. The limits of the annealing range within which

this grain-growth will be produced are 650 deg. and 900 deg. C.

4. When the strain is less than a certain or "critical" amount, the annealing range seems to be limited by the thermal critical points at about 690 deg. and 780 deg. C.

5. The most practical standard by which to measure the permanent deformation seems to be the reduction of area. No grain-growth occurs following a reduction of area of less than about 7 per cent or more than about 25 to 30 per cent. The "critical" strain seems to be marked by a reduction of area of about 9 per cent.

6. The refining action taking place at about 780 deg. C. is a further indication of the presence of a thermal critical point at this temperature.

7. The presence of carbon causes a suppression of grain-growth, an effect which, while negligible when the carbon content is low, is very pronounced when the steel contains 0.15 per cent or more. No noticeable grain-growth was found to occur in steels which were uniformly above 0.18 per cent in carbon content.

Grain-growth can therefore be avoided by the application of comparatively heavy work to the metal before annealing, or by proper control of the annealing temperature. If the cold-worked metal is not annealed no grain-growth can occur.

The president, Sir Robert Hadfield, in presenting the paper, said that experiments he was carrying out showed that far below 300 deg. C. phenomena occurred in steel showing it not to be in a quiescent state. This fact was not properly appreciated, and it might explain why breakages often took place long after the material had been hardened. He wished Mr. Sherry had not confined himself to only one type of steel.

Dr. W. Rosenhain said Mr. Sherry's paper gave a new significance to the importance of "ghosts" in mild steel. In these relatively carbonaceous areas grain-growth could occur, and if this was facilitated by phosphorus the handling of mild steel needed careful watching. The annealing point recommended (650 deg. C.) was only safe in a material that did not rely on carbon content for the greater portion of its strength. It could be inferred from the paper that the theory of the existence of a thermal critical point at about 780 deg. was far from dead.

**Specific Resistance of Iron.**—At the eighty-first meeting of the Faraday Society in London, Dec. 18, 1916, Prof. E. D. CAMPBELL of the University of Michigan, U. S. A., presented a paper entitled "Do Equiatomic Solutions in Iron Possess Equal Resistances?" The following is an abstract of the paper, which was read for the author by Sir Robert Hadfield:

The conception of steel as a solid solution has long suggested a relationship between its chemical composition and resistance. Le Chatelier's work in 1898 indicated that equiatomic concentrations of carbon as hardening carbon and silicon exerted practically equal effect on the specific resistance, while that due to manganese was only about five-sevenths as great, that of nickel three-sevenths to five-sevenths as great, and that due to chromium, tungsten, and molybdenum was very slight. Benedicks, in 1902, laid down the general law that equiatomic solid solutions in iron possess equal resistances.

The experimental work of Arnold has shown the assumptions underlying Benedicks' law to be untenable, and the object of the author's experimental work was to seek a more satisfactory hypothesis. The experiments, which are described in the paper, were carried out on seven steels of varying compositions, and their specific resistances were measured in both the hardened and annealed states. The deviations from the calculated values cannot be explained on Benedicks' as-

sumption, but they suggest that it is the *molecular* concentration of the *carbides* in solid solution and not the *atomic* concentration of the *carbon* which determines the influence on the specific resistance exerted by such solutes.

The almost constant value of the absolute increase in specific resistance with increased temperature, this increase differing very slightly from that of pure iron, would indicate that the increase is almost wholly due to the increase in specific resistance of the solvent, while that component of the total specific resistance due to the solutes in solid solution is only slightly affected by the temperature at which the specific measurements are made.

#### Tungsten

**Manufacture of Tungsten in England.**—When the war broke out the manufacturers of high-speed tool steel in England found themselves in much the same position as the users of dyestuffs. The *Engineer* (London) states that, following a Government inquiry into the manufacture of tungsten, a committee was formed of representatives of leading steel makers and thirty-one firms took shares in a company which was formed, called High Speed Steel Alloys, Ltd. The erection of the works was commenced about the end of 1914, and by July, 1915, the production was begun. In September, 1915, the British Government took control of all wolfram ore within the British empire and divided it among the makers of tungsten and ferro-tungsten. To assist in increasing the output of ore within the empire the company purchased mines in Burma, which have been placed under the control of Dr. W. R. Jones, who was formerly in charge of the Indian Survey. Ore is also obtained from Australia, and it is hoped that before long South Africa will be another source of supply. The works of the company are now in full operation and are using the following process: The ore is crushed, ground, screened and mixed with soda. A magnetic separator is used for separating tin ore from the tungsten ore. The mixed ore and soda are roasted in coal-fired reverberatories, requiring two to three hours. The furnace product is boiled with water and the tungstate of soda dissolved. The solution is then filtered and the filtrate containing the tungsten is treated with hydrochloric acid, when tungstic oxide precipitates. This is filtered off and dried, mixed with powdered anthracite coal and placed in crucibles, which are heated in coke stoves. The last-named process will, however, shortly be improved upon as a new continuous furnace, heated by gas from two Wilson bituminous gas producers, is now being erected. The reduced metal is then washed in dish-shaped trays and dried, yielding the final product in the shape of a fine chocolate colored powder of 98 to 99 per cent purity. This is packed in tin-lined wooden boxes containing about 200 lb. ready for delivery.

#### Refractories

**A Carbon Tube Furnace for Testing Refractories.**—At the eighty-first meeting of the Faraday Society, held in London, Dec. 18, 1916, EZER GRIFFITHS and EDGAR A. GRIFFITHS of the National Physical Laboratory, described and exhibited a carbon tube furnace for testing the softening points and compressive strengths of refractories.

The paper describes a carbon tube furnace designed for the testing of refractory materials under definite load.

The specimens are cut from the brick and ground up into the form of short cylinders. Pressure is applied by means of springs suitably connected to carbon rods which carry the specimen under test.



Two simple forms of electrode construction are described. In one of them the current is carried by two copper tubes bent into a zig-zag form and cast into two blocks of white bearing metal. The faces of the blocks are cast to the form of the carbon tube to which they are clamped. The copper tubes also serve for water cooling.

The temperature of the specimen is directly observed by means of a polarizing type of optical pyrometer. For this purpose a slot  $\frac{1}{4}$  in. wide extends the entire length of the tube. A slot was made in preference to a round hole, since the latter would have produced a discontinuity in the resistance of the tube.

In the discussion Dr. W. Rosenhain remarked that the results obtained depended upon the surfaces between the little columns being absolutely true. In the case of a material which softened with temperature the results would be affected by the intensity of the pressure.

Mr. H. M. Ridge said he was glad to know of an apparatus that could be subjected to over 1500° C. and at the same time give accurate tests of firebrick materials. He hoped that if comparative tests had been made on the standard bricks they would be published. The recent regulation fixing a maximum price for firebricks militated against the good brick, but enabled the maker of inferior bricks to raise his prices. The use of an apparatus like that described would enable a sliding scale to be fixed on a basis of actual tests.

Dr. H. Borns asked how long a test took.

Dr. H. C. Greenwood asked whether tests had been made on the effect of temperature on tensile strength.

Dr. R. Lessing thought the test pieces used in the furnace rather too small, especially for large-grained material.

Mr. E. Griffiths, in reply, said there was no difficulty in applying the pressure normally. Usually three specimens were taken from a brick and the results only accepted when concordant. A small test-piece ensured uniform distribution of temperature. A test usually took  $2\frac{1}{2}$  hours at a 10° C. increase of temperature per minute. Comparative tests could not be published at present, but he might say that a magnesite brick broke down at 1500° C. when heated uniformly, under a pressure of about 30 lb. This was about the normal.

#### Electrochemistry

**Electrochemical Analogies of Photochemistry.**—At the Cleveland meeting of the American Physical Society, Oct. 27 and 28, 1916, WILLIAM ROY MOTT of the National Carbon Company's Research Laboratory, Cleveland, Ohio, read a paper on "Electrochemical Analogies of Photochemistry." An abstract was published in the *Physical Review* for January, 1917. The author has made experiments on dye fading and other photochemical reactions and has come to the conclusion that the general theory of the electrochemistry of photochemistry can be revised to a more concrete form than expressed by the theory of Grotthus. In the revised theory the two electrochemical factors of current and voltage are respectively related to amount of light and wave-length of light.

He first discusses the analogy of current and amount of light, citing Faraday's law and the photochemical law, that the amount of chemical action is nearly proportional to the energy of the light absorbed in the photochemical reaction chamber. The limitations of these laws, viz.: the necessity for a certain minimum voltage in Faraday's law and the wave length limits in the photochemical law are discussed.

The great importance of absorbing media in photochemistry leaves no apparent relation to electrochemical equivalents as in electrochemistry in going from

one material to a different material. The departures from apparent proportionality can be explained, however, in a manner similar to the apparent departures of electrochemical reaction from Faraday's law in a given electrolyte.

In developing the analogy of voltage and wave-length the author considers the wave length as an intensity factor of light corresponding to voltage as an intensity factor of electricity, and cites examples showing how the shorter wave length light has a greater chemical effect. The following table illustrates the generalization:

Material	Formula	Voltage for Decomposition	
Calcium fluoride...	CaF <sub>2</sub>	4.7	Most transparent to far ultraviolet.
Hydrofluoric acid...	HF	1.2	Not decomposed by far ultraviolet.
Hydrochloric acid...	HCl	1.7	Slightly decomposed by far ultraviolet.
Hydrobromic acid...	HBr	1.2	Easily decomposed by ultraviolet.
Hydriodic acid...	HI	0.6	Easily decomposed by blue light.

A curve could probably be constructed between wave length of light required and decomposition voltage required. It is interesting to note that the extremely short waves are readily absorbed by everything except the non-reactive noble gases.

This theory can be further expanded to show a relationship between heat of formation (on electrochemical equivalents and refractive index and point of probable absorption of light as the curve is carried into ultraviolet region. Also, the theory has fascinating possibilities in predicting the character and probability of light production by chemical change.

#### Glass

**Union of Glass by Heat Treatment.**—A paper on "The Union of Glass in Optical Contact by Heat Treatment" was presented at the Faraday Society meeting in London, Dec. 18, 1916, by R. G. PARKER and A. J. DALLADAY. The paper gives the results of some interesting experiments on joining glass.

Two pieces of glass in optical contact can be made to join completely, and become one piece, by carefully controlled heating. The temperature employed is very far below the melting point, being 80–90° C. below the annealing point; the glass components therefore unite without softening and without deformation of unsupported optically worked surfaces. Slight pressure is usually applied. The components, after treatment, are not separable by any of the means which suffice to separate pieces in optical contact, viz., by liquids (capillarity), by sudden small temperature changes, or by mechanical force. When subjected to the last of these, or cut with a diamond, a composite block breaks as would one piece. The accuracy of the parts is unaltered by the heating, and it is not difficult to prepare by this method cells having windows parallel to one second, with optically worked surfaces in the interior. Within limits, different kinds of glass can be joined; their expansion coefficients must not differ greatly, nor must the annealing points differ by more than 50° C.; they must also be kinds which will fuse together in a blow-pipe flame. Only when the components are all of one kind of glass can the composite article be freed from strain by annealing. Fused silica plates unite at 1030° C.

Dr. W. Rosenhain regarded the authors' results as a remarkable achievement. He thought that various methods of polishing might make a difference in the behavior of the glass. He discussed the behavior of moisture or hydrated films as affecting the union.

Might not chemical cleaning give better results than mechanical?

Professor Alfred W. Porter thought the fact that the outside surface film of the glass was in a totally different condition from that of the inside was responsible for the possibility of the process. The outside film would be practically in a flowing state.

Mr. Dalladay, in replying said chemical cleaning might be useful, but it had not when tried given good results. Such trouble as occurred was caused by little pieces of cellulose left from the material used for wiping the surface.

#### Transportation

**Transport of Corrosive Liquids by Water.**—The transportation of acids and other corrosive chemicals has always been a difficult problem. In the *Journal of the Society of Chemical Industry* for December 30, 1916, H. N. MORRIS describes a method of transportation in a tank boat, following out the ideas of the oil tankers developed 10 years ago by the Standard Oil Co. He reviews the common methods of transporting acids at sea and shows that they are in general costly and dangerous to the ship, although cases are known where no accidents have occurred for many years. At the best the methods are, however, very costly. The author describes a proposed design for what he terms a "sub-aqueous acid tank," consisting of two shells or tanks, one within the other. The inner tank, which carries the corrosive liquid, may be suitably lined with lead, aluminum, bitumen, or materials depending on the liquids to be transported. By towing this tank, a safe and economical means of transport is provided. A light on the tank is required to safeguard against loss.

A tank of 250 tons of ordinary conc.  $H_2SO_4$  is described, which could be used equally well for oleum, which would solidify, but could be easily melted by passing steam into the outer case until the contents of the inner tank were liquid. In this case it would be better not to line with lead, but to use stronger iron.

It could also be used for hydrochloric acid if the lead lining were covered with a uniform coating of bitumen. It could be used for the conveyance of nitric acid if lined with aluminium. As the inner tank is divided into three compartments three different acids or corrosive liquids could be transported at once.

In many cases a return cargo of oils or other liquids could be obtained. The tank is not suitable for very small quantities, but for short distances, 100-ton or even 50-ton tanks might be used economically, and there are not many countries where it would not be possible to dispose of 250 tons of either sulphuric acid or mixed consignments. The tank can be used for many acids and other chemicals: sulphuric acid, oleum, hydrochloric acid and nitric acid, carbon bisulphide, alcohol, benzol, toluol, or naphtha, which, although they cannot all be classed as corrosive, are more or less dangerous for a steamship company to carry.

**New Swedish Nitric Acid Company.**—A new company with large capital has been formed for the manufacture of nitric acid and other chemical products in Sweden, according to the *Chemical Trade Journal*. It has entered upon a contract with the Royal Waterfalls Board for a supply of electric energy, and the works will be erected on the area reserved for industrial installations at Trollhattan. The annual capacity will be some 7000 tons concentrated nitric acid, with nitrates as an auxiliary product. The new company will use the Birkeland-Eyde method, for which rights have been secured for Sweden. The work is being pushed ahead with all speed to be ready this year.

## Recent Metallurgical and Chemical Patents

### Copper

**Hydrometallurgy of Copper.**—A patent was granted to EDWARD RAY WEIDLEIN, of Thompson, Nev., for the extraction of copper from its ores by means of sulphuric acid and the precipitation of the copper from the sulphuric acid by means of sulphur dioxide, according to the equation,  $CuSO_4 + SO_2 + 2H_2O = Cu + 2H_2SO_4$ .

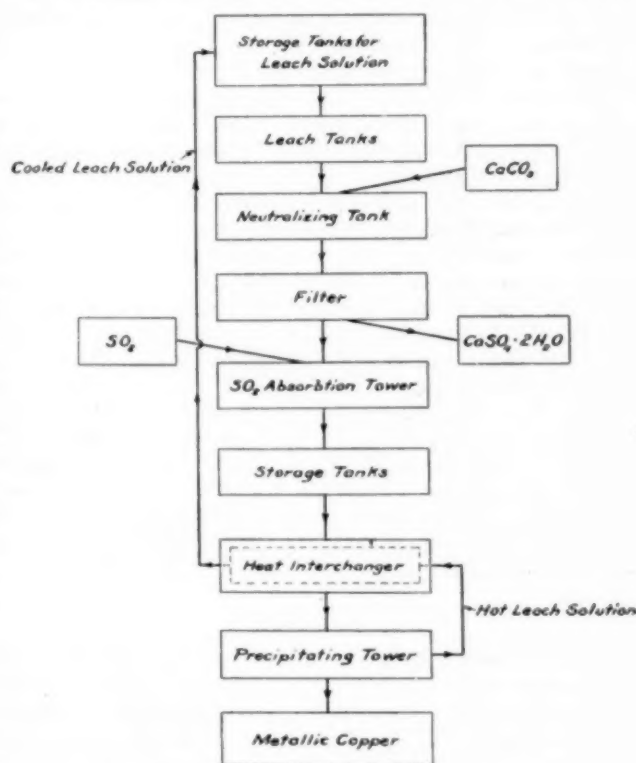


FIG. 1—PROCESS FOR EXTRACTING COPPER

Fig. 1 represents the flow sheet of the process. In preferred practice the ore is leached with a 3.6 per cent sulphuric acid solution, the solution is then neutralized and the copper sulphate solution containing about 1.5 per cent copper is treated with an approximately equal amount of sulphur dioxide. The solution is then heated in the precipitation tanks to 150 deg. C. at a pressure of about 100 lb. per square inch. Under these conditions the copper will be precipitated. It is removed by filtration, and the solution is re-used for leaching. The process, it is claimed, may be used on ores, mattes and other copper bearing materials. (1,201,899, Oct. 17, 1916.)

**Method of Connecting Electrodes in Electrolytic Refining.**—ROBERT L. WHITEHEAD of Perth Amboy, N. J., has been granted patents on a method of arranging the electrode connections in a series of electrolytic vats, and on a method of making the individual contact. The method was developed in connection with the electrolytic refining of copper. The patents have been assigned to the American Smelting & Refining Company of Maurer, N. J. The original application was filed in February, 1913, containing claims to both the form of contact and the manner of arranging the connections. The application was subsequently divided and these two claims presented in separate applications. The method of arranging the connections is to connect the cathodes of one vat directly to the anodes of the adjacent vat, instead of as heretofore having two sets of contacts along the common bar supporting the cathodes of the first vat and the anodes of the second vat.



By connecting the cathodes of one vat directly to the anodes of the second vat the common busbar is dispensed with and the number of contacts is reduced to one-half. The connection, which is the subject of a second patent, is made by cutting a V-shaped groove in one electrode and making a projection on the other electrode to fit into this groove (1,206,963-65, Dec. 5, 1916).

#### Alloys

**Nickel-Chrome Alloy.**—An alloy of high melting point and high resistance to the dissolving action of acids and alkalis has been patented by THEODORE B. BRIX of Newark, N. J. It consists of not less than about 55 per cent of one of the metals of the nickel group, from 15 to 35 per cent of chromium, chromium and tungsten or chromium and titanium and not over 10 per cent of silicon. Usually nickel, chromium and silicon are the constituents used, but for certain purposes, cobalt may in part replace the nickel, or combinations as mentioned may replace the straight chromium. In varying the metals, high resistance to temperature, insolubility in acids and alkalis, malleability or ductility may be attained. Other metals such as copper, boron, aluminium and manganese may be used in small amounts. The preferred alloy, embodying practically all the desired properties, is given as from 60 to 75 per cent by weight of nickel, 15 to 20 per cent of chromium, 5 per cent copper, 4 per cent silicon, 1 to 4 per cent tungsten, 2 per cent aluminium, 3 per cent manganese-titanium ( $\frac{2}{3}$  manganese) and 1 per cent boron. A method for the production of said alloy is described. (1,203,180, Oct. 31, 1916).

#### Sintering

**Method and Apparatus for Sintering Metals.**—An apparatus for the sintering of metallic powders, such as tungsten, has been patented by RAYMOND B. WALLING of Newark, N. J. Fig. 2 represents the apparatus employed. The apparatus proper is enclosed in a copper treating bottle, cylindrical in shape. The bottle is provided with a jacket 2. Between the jacket and the

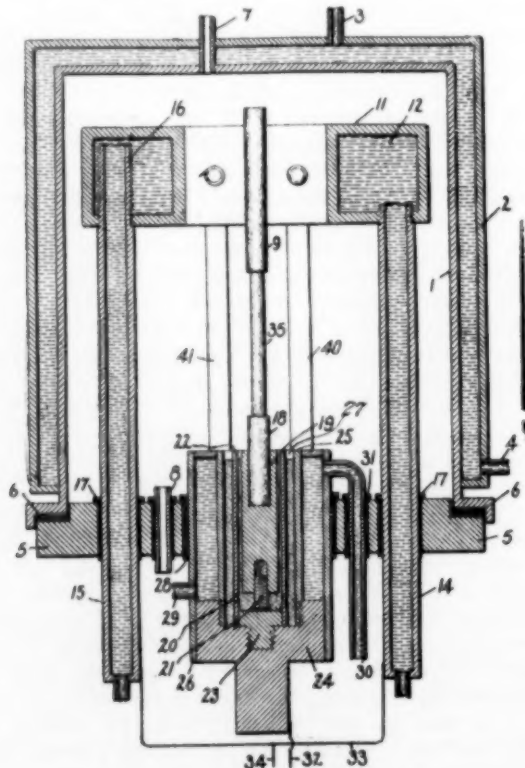


FIG. 2—SINTERING APPARATUS

bottle is a space for the circulation of a cooling medium, such as water, which enters at pipe 4 being discharged through 3. In its lowered position the bottle rests on plate 5 and is rendered gas-tight by gasket 6. During the process of operation, hydrogen gas or some other inert gas is circulated through pipes 7 and 8; 9 is the upper electrode and is made of the material which is to be sintered (in this instance tungsten), the electrical contact being made with copper clamps 10 and 11. Cooling arrangements are provided for the electrode holders; 18 is the lower electrode, and is firmly set into the copper casting, which floats in mercury bath 20; 21 is a screw which regulates the height of the mercury and also the downward motion of the electrode 18. Cooling devices are also described for this electrode. 35 represents the tungsten rod which is to be sintered by means of heat generated by the resistance of the passage of an electric current through the rod. The main features of this apparatus are the use of the electrical current and the floating mercury contact for the lower electrode. It is claimed that by using this method there is no waste whatever, as the rod is perfectly sintered throughout its entire length (1,194,906, Aug. 15, 1916).

**Apparatus for Roasting and Sintering Ores.**—A patent granted to JAMES GAYLEY of New York, N. Y., involves improvements made on the Dwight-Lloyd type of sintering machines. The main feature of the improvements consists in making the joints between the moving pallets and the wind box automatically tight, thus preventing the impairment of the airtight union between the two. Fig. 3 represents the longitudinal

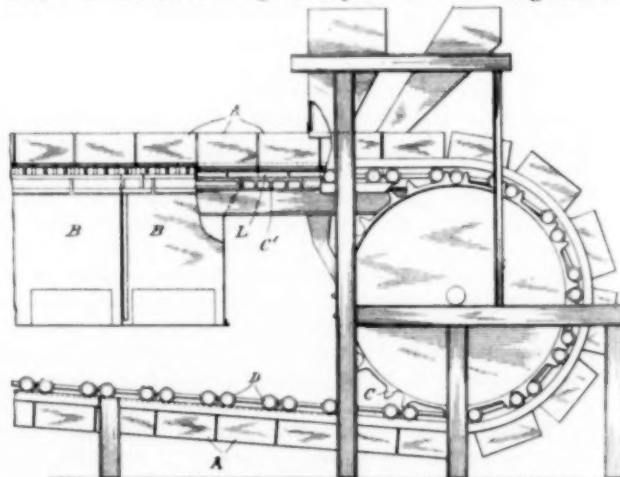


FIG. 3—ROASTING APPARATUS

section of a part of the machine and Fig. 4 is a vertical cross-section of the same. A train of pallets A moves continuously over the top of the wind box B. The means of locomotion of these pallets is similar as in the Dwight-Lloyd machine. The whole length of the wind box top is fitted on either side with a rectangular wearing bar F, and at each end it is filled with a dead plate

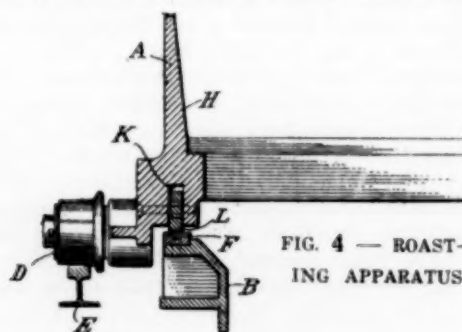


FIG. 4 — ROASTING APPARATUS

C', over which the whole width of the pallet slides to prevent the leakage of air at these points. The wearing bars constitute the fixed member of the sliding joint which is maintained between the pallets and the wind box. Bars or plates fit in grooves K in the under surface of sides H. These bars or plates are termed curtains and are designated as L. In their simplest form these curtains consist of rectangular plates or bars, having the same length as the pallets. These plates or bars fit into the recesses in the pallet with only sufficient clearance to permit free movement of the pallets under their own weight. M are rods or pins which prevent the endwise movement of the curtains in the pallets. These improvements, it is claimed, give the desired automatic airtight joint (1,197,199, Sept. 5, 1916).

## The Utilization of Waste-Products and of Low-Grade Fuels for Power-Generation

By John B. C. Kershaw, F.I.C.

### TOWNS' REFUSE

The rising cost of all classes of coal and slack due to causes, some permanent and some we may hope only temporary in character, but which it is unnecessary to analyze here, renders the discussion of the utilization of waste materials and low-grade fuels for power purposes of particular value at the present moment. The power engineer has been badly hit by the increase in the cost of fuel, and since it is highly probable that a large share of this increase will prove to be permanent, it is necessary that he should take a wide survey of all combustible materials, and impress into his service any that promise to yield a fair return for their cost of collection and cartage, as fuel for his plant.

Foremost among the waste materials that may be utilized as substitutes for ordinary fuel, is that of towns' refuse, since not only is its quantity large, but its destruction by burning is imperative in all large towns and crowded centers of population. Further, the heat produced by its combustion is most easily utilized by means of steam boilers and electrical generators, and the slag produced by its combustion may also prove a valuable source of revenue.

In this article the writer proposes to discuss:

1. The published data relating to the calorific value of this refuse for steam raising purposes.
2. Recent improvements of the destructor process.
3. The practical value of refuse destructors from the standpoint of the power engineer.

#### 1. THE CALORIFIC VALUE OF TOWNS' REFUSE

It must be admitted that many of the published figures relating to the calorific value of towns' refuse are unreliable and misleading and that some of the earlier estimates of the number of kilowatts that might be produced from a ton of refuse were much too high, and have only been approached in specially situated districts where the calorific value of the refuse is unusually good.

Towns' refuse is a product that varies greatly in composition and value, not only in different towns, but also in the different districts of the same town, and at different periods of the year. The only figures that are reliable as to its calorific value therefore, are those based upon twelve months' operation of the destructor plant, since the summer value of the refuse is generally lower than the winter.

In colliery districts where the miners' families obtain their coal for nothing, or at a nominal price, and the general public pay only about one-half of the price charged to the consumers in large towns, the fuel value

of the refuse will also be twice as high, owing to the presence of much unburned carbon in the cinders.

In towns and cities far from coal-mining districts on the other hand, the refuse will be found to consist largely of kitchen and garden garbage, which, if wet, has little value for steam-raising purposes.

Bearing these facts in mind, one can now proceed to examine and analyze the figures obtained in tests of the more recently erected destructor plants, with a view to ascertaining the real fuel value of the refuse collected from a town of average size. The figures presented are mostly those obtained during test runs of the plant when taken over from the engineering firm which designed and erected it, and a standing deduction of 33.3 per cent will be made from these figures, in order to bring them into line with the results that may be expected from twelve months' practical operation of the plant. This percentage deduction has been arrived at by study of the comparative data published in the early years of destructor development for short test runs, and for one year's operation of combined destructor and electric generating plant.

#### THE HORSFALL DESTRUCTOR

"The New Destructor Company," who now control the Horsfall system of refuse destruction give, in a recent publication, a tabular statement showing the gradual improvement in the efficiency of their destructors during the period 1900-1910. The results are based on tests of not less than twenty-four hours' duration, and the water evaporated per pound of refuse shows an increase from 1.21 lb. for the destructor erected at the Pembroke Electricity Works, Dublin, to 1.57 lb. for the Poplar Electricity Works, in 1910. The details of this latest available test for the Horsfall type of destructor are as follows:

Place—Poplar.

Date of test—Feb.-March, 1910.

Tons of refuse burned per twenty-four hours—153 tons.

Evaporation per pound of refuse at 212 Fahr.—1.57 lb.

The adoption of closed tubes with hopper bottoms for storing the refuse at the destructor plant until charged into the furnaces, and of a mechanical clinkering grate in the form of a tipping truck, are the latest improvements of the Horsfall Destructor.

#### THE HEENAN AND FROUDE DESTRUCTOR

The following results of recent tests with the Heenan and Froude type of refuse destructor are of special interest, since they emphasize the great variation in the thermal value of the refuse in different parts of the country, and illustrate the remarks made on this point in the introduction to this article.

Town	Date of Test	Period of Test	Lbs. of Water Evaporated per Lb. of Refuse
Aberdare (S. Wales)	March 24, 1915	10 hours	3.53
Pontypridd (S. Wales)	Feb. 10, 1910	8 hours	2.69
Coventry	April, 1911	48 hours	2.00
Rotherham	Jan. 27, 1910	18 hours	1.80
Clydebank	May 2, 1907	8½ hours	1.73
West Bromwich		8½ hours	1.45
Swinton	Sept. 24, 1912	11 hours	1.32
Cheltenham	June 26, 1908	17½ hours	1.24
Rotterdam	Feb. and May, 1913	6 days	1.10
Rotterdam	Feb. and May, 1913	10 days	.88

N.B.—These are actual evaporations, and are not expressed on the standard basis "from and at 212 deg. F."

The adoption of water-sealed charging devices, and of improved methods of withdrawing and quenching the clinker, are some of the latest improvements in the Heenan and Froude type of destructor.



## FOREIGN TYPES OF DESTRUCTOR

The first destructor erected in Germany was that at Hamburg in 1896, a result of the cholera epidemic in the previous year. This was of the Horsfall type, and some trouble due to the use of steam jets was experienced before it worked satisfactorily. Brussels followed the example of Hamburg in 1903, and Zurich in 1904, by erecting similar destructor plants. These were also of the Horsfall type. The first German-designed destructors were those erected at Wiesbaden by Dörr, and at Kiel by Herbertz, in 1905-1906. These designers made use of the principle of using hollow chambers in the walls of the destructor cell for the purpose of heating the incoming air, which was forced by fans into the combustion chamber under a pressure of 400 mm. (16 in.) water.

As might be expected from the methods of domestic heating in use in Germany (by closed stoves using chiefly briquette-fuel) the refuse collected in German towns and cities is less rich in combustible material than English refuse. At *Puchheim*, the evaporative efficiency is 0.891 lb. of water per pound of refuse, (equivalent to 1.01 lb. from and at 212 deg. Fahr.) while at *Fürth* (Humboldt system) the actual evaporative efficiency is 0.98 lb. water per pound refuse, equivalent to 0.97 lb. from and at 212 deg. Fahr. The general results obtained at these two installations show that German refuse has an average heat value of only 2160 B.t.u. per pound, and that about one-half of this heat value can be utilized for steam-raising purposes.

The English figures, consequently, show much higher evaporative efficiencies, the average for the Heenan and Froude type of destructor being over 1.87 lb. water per pound of refuse and that for the Horsfall destructor being 1.38 lb. Taking 33.3 per cent off these figures we have 1.25 and 0.94 lb., respectively. It is unlikely, however, that in view of the increased price of fuel in recent years and the urgent necessity for the practice of economy on the part of all sections of the population, that English house refuse will in future contain so much cinder and unburned carbon as in the past, and it will be wiser, therefore, to assume that the average calorific value of towns' refuse, when calculated upon twelve months' working of the destructor, is about 2160 B.T.U per lb., and that only 1 lb. of water can be evaporated per lb. of refuse burnt, for steam-raising purposes. The practical application of these figures will be discussed in the third portion of this article, when discussing the generation of electricity from house refuse.

## II. RECENT IMPROVEMENTS AND THE CONTROL OF THE DESTRUCTOR PROCESS

The difficulties met with in the destruction of house refuse by burning are chiefly those arising from the physical character of the materials to be consumed, and the large amount of clinker produced as a result of the combustion of the refuse. Kitchen garbage and similar refuse is generally wet, and will not burn at all until dried, while the rags, waste paper and cinders, that form the other chief combustible elements of the refuse, are so distributed and buried among the mass of incombustible material that it is very difficult to get an adequate air supply at the points where it is most required. According to Broadbent, the average composition of London refuse is as follows: Ash, 47 per cent; breeze and cinder, 25.5 per cent; paper, straw and vegetable refuse, 13 per cent; dust and dirt, 9.75 per cent, while the remaining 4.75 per cent is made up of bones and offal, rags, bottles, crockery, broken glass and tin cans. The combustible material, therefore, does not amount to more than one-third of the whole, and

the difficulties of burning it are much greater than those encountered in burning the poorest qualities of slack or coke breeze containing 30 per cent to 35 per cent of ash.

When the combustion has been successfully effected the fused clinker that remains behind is over one-third the weight of the original charge of refuse, and special forms of grate and special appliances are necessary in order to deal with this large amount of molten matter and to avoid heat losses by its withdrawal from the furnace.

The difficulties of attaining complete combustion have been overcome by the use of fans for supplying preheated air under considerable pressure, perforated iron plates being employed for the furnace sides and bottoms in many of the newer types of destructor in order to distribute this air through the whole mass of material to be burned. The air is preheated either by passing through the hollow well spaces between the different cells of the destructor or by special air heaters placed in the hottest part of the combustion chamber of the furnace. It is now recognized by designers of destructor cells that a preheated air supply is a *sine qua non* of good destructor working, for a difference of 300 deg. C. in the temperature of the incoming air makes a difference of 350 deg. to 400 deg. C. in the actual temperature attained in the furnace. The advantages of a preheated air supply are not confined to this increase of thermal efficiency, for a quicker ignition of the refuse and an accelerated combustion accompany the use of hot air, and the capacity of the destructor per day of twenty-four hours is therefore greatly increased.

As regards the improved methods of dealing with the clinker, it has been found by actual measurements that the heat retained in the fused mass only amounts to from 3 per cent to 4 per cent of that generated, and attempts to utilize this heat for raising the temperature of the incoming air or for other purposes have not proved so successful as was hoped. The use of water-cooled mold-shaped grates which facilitate the removal of the clinker in one mass and of a bar bent in a spiral form around which the clinker melts are the latest devices for dealing with the clinker problem, since by means of a ring-shaped handle to the clinker bar and a winch a mass of fused clinker weighing several hundredweights can be removed from the furnace with ease by one man.

As regards charging the refuse into the destructor, the double-bell system has been improved upon, a whole charge now being dropped into a water-sealed pocket in one operation before the lower bell is raised and the charge is passed on into the furnace.

The latest development of destructor design, however, is the adoption of the blast furnace as the model of what a refuse destructor should be, the Dörr destructor erected at Wiesbaden being the first of this type. Improved forms of the shaft type of destructor have been erected at Barmen and Hamburg, and as these represent a distinct departure from the ordinary type of destructor design, and can be operated with reduced costs, a few details of their construction and method of working are appended. The original Hamburg furnace was constructed in the form of an octagonal shaft, the hearth of which was rectangular in form and water-jacketed for cooling purposes to prevent the clinker adhering to the walls. A perforated iron gating was used for distribution of the air supply, and a fan supplied air under pressure to the air chamber below the grating. This furnace gave a mean temperature of 1050 deg. C. and burnt successfully 31 tons of refuse per day.

An improved form of the Hamburg shaft furnace

was erected later at Barmen. In this case a preliminary grate was provided in front of the grating of the shaft furnace, and on to this the clinker cake was drawn and remained twenty to forty minutes, in order that it might give up some of its heat to the incoming air. As already stated, the heat losses with the clinker are small and the temperature of the incoming air cannot be raised to any considerable extent by contact with it.

The latest types of the shaft destructor furnace have, therefore, adopted special air heaters placed in the hottest part of the system for preheating the air supply, and only use the clinker heat for the preliminary step in the preheating process.

Kohlmann,\* in a valuable paper upon the "Evolution of the Refuse Destructor," read before the Canadian Section of the Society of Chemical Industry in 1913, has given the following figures for the capacity of the various types of destructor, and if these can be accepted as reliable the superiority of the shaft type of furnace with preliminary air heating is amply demonstrated. The fact that with this type of furnace the labor costs for charging and removing the clinker are also reduced to a minimum would indicate that in time we may expect to find all dust destructors approximating to this type of design.

System	City	Grate Area Sq. Metres	Capacity per Hour per Sq. M. of Grate
Horsfall	Hamburg (old)	2.75	{ 76 x133 x220
	Zurich	1.80	133
	Brussels	2.5	133
Heenan	Milwaukee	2.5	335
	Dublin	2.7	260
	Cheltenham	2.7	235
	Vancouver	3.0	255
	Coventry	2.7	385
Herberts	Fiume	1.25	345.75
	Brünn	1.25	325
	Kiel	0.94	445
	Frankfurt	1.1	285
		2.2	480
Humboldt	Fürth	1.5	720
Dörr	Wiesbaden	1.0	815
Hamburg	Hamburg (new)	1.2	1330

### III. THE PRACTICAL VALUE OF REFUSE DESTRUCTORS FROM THE POWER ENGINEER'S STANDPOINT

The figures given in the first section of this article prove that house refuse has an average heat value of one-seventh that of ordinary coal, and that about 1 lb. of steam can be produced per pound of refuse burnt. Has such a poor fuel any value at all from the power engineer's standpoint? If this question were decided solely upon the value and merits of this refuse as a fuel, the answer to the question would be in the negative, for the interest upon the capital outlay on the destructor plant and the costs of working it would amount to more than the value of the steam produced. But, as pointed out in the introduction to this article, the question of refuse destruction by burning is fundamentally a sanitary one. All large towns and cities ought to be provided with destructors with sufficient capacity to cremate the whole of their refuse, and the question of utilizing the surplus heat to the best advantage is really an economic one, bound up with the successful operation of these plants.

In the early days of destructor development it was customary to erect and work these plants as adjuncts of the electricity supply stations, and it was claimed that from 40 to 60 units of electricity could be obtained in these combined plants per ton of refuse burnt. The

L. S. D. value of the latter as fuel was calculated therefore to be 3s. 4d. (\$0.80) per ton.

The electrical engineers in charge of large power-generating stations, however, were not in favor of the system, since a certain amount of nuisance and dust detrimental to the efficient working of the machinery arose from the carting and tipping of the refuse, and only a few of these combined plants have been erected in recent years.

The modern plan is to erect the destructor on the site most suitable for the collection of the refuse, to operate it as an independent works, and to sell the steam produced by the surplus heat to the nearest factory or works at an agreed rate per 1000 lb. In some cases this sale is to an electricity supply station, but this is not an essential feature of the plan, and any large works that requires steam for heating or power purposes might cover a portion of its requirements from a destructor plant if one were near at hand. As examples of this system of working, the destructor plants at Huddersfield, England, and at Rotterdam, Holland, may be referred to.

The Huddersfield destructor was erected in 1910 by Manlove & Alliot of Nottingham, and consists of eight furnaces or cells, arranged in two units of four cells each, each unit being complete with its own combustion chamber and water-tube boiler. The cells are of the "back-feed" type, and are arranged on the continuous system. Each cell has a grate area of 25 sq. ft. and is provided with a drying hearth at the rear, where the green refuse is subjected to a preliminary drying. Forced draught is provided by two electrically driven fans, and the air is preheated before its entrance into the combustion chamber. The surplus steam not required for the operation of the destructor plant is carried by well-insulated steam mains over a roadway to the electricity works, and is sold to this undertaking at a net cost of 7d. (14 cents) per 1000 lb. The annual revenue received by the destructor plant from this source in the four years 1911-1914 has been as follows: 1911, \$7,525; 1912, \$8,795; 1913, \$8,950, and 1914, \$8,505.

The Rotterdam plant was erected in 1911, and was first put into operation in 1912. The destructors are of the well-known Heenan & Froude type, and are provided with all the improvements brought out by this firm in recent years. The plant consists of five independent units, each consisting of two sets of four cells, grouped on either side of a central combustion chamber. The grates of four adjoining cells are separated from one another by low cast-iron transverse dead-plates of hollow construction, so that the four grates practically form one continuous furnace chamber.

Each unit is fitted with a "regenerator" consisting of a group of vertical boiler tubes expanded at the top and the bottom into horizontal tube-plates. The hot gases from the boilers pass through the tubes and down into dust pits, while the draught air passes over the outside of the tubes.

The most striking feature of the installation, however, is the system of collecting the house and street refuse of the city and of bringing it to the destructor furnaces with a minimum of nuisance and cost. The city is divided into five districts, in each of which there is a small dock for accommodating barges. To these docks the refuse is brought in tip-carts, which tip the refuse direct into skips, a number of which fit neatly into a barge. The refuse never leaves these skips until it goes into the furnaces. Each barge holds about 2 dozen skips and each skip contains about 1600 kg. (31.5 cwt.) of refuse. The skips have bottom doors, which can be tripped when the device is lowered into its seat-

\*Journal Soc. Chem. Industry, April 30, 1914.



ing over the furnace; also they are made larger at the bottom than at the top, so that the rubbish will fall out quite readily.

It is chiefly in this respect that the older systems of destructor working were faulty and capable of improvement, and although the possession of canals that ramify all through the city simplifies the collection and transport of the refuse in Rotterdam, there is no doubt that many of the good points of the Rotterdam system might be copied with considerable success in other towns and cities.

The official trials of the plant, made during separate runs in February and May, 1913, showed that 1214 tons and 1228 tons of refuse had been cremated, with an evaporation of 1.106 lb. and 0.889 lb. of water per pound of refuse burnt respectively, an average of 144 tons per day, and 0.99 lb. steam for the whole seventeen days' period of trial. The steam raised is used partly in the plant where the clinker is crushed and graded to three sizes, and all iron is removed by magnetic separators, while the surplus is sold to the municipal electricity supply department at the fixed rate and is used for driving a 1250-kw. turbo-generator.

The city electricity department provided half the capital required for the electrical portion of the plant and for the building in which it is housed, pays the whole of the running charges of the same, and 0.10 penny (0.20 cent) per unit for the steam. Further details of this interesting plant will be found in the descriptive article from which these facts are taken, which appeared last year in the publication referred to below.\*

The one disadvantage of the Rotterdam system of working appears to be that there is no opportunity for sorting the refuse and removing from it the paper, old metals and broken glass, all of which have a marketable value, especially at the present time.

As an indication of the annual revenue that may be derived from this source by a destructor plant, the following figures from the 1914 report of the chief sanitary inspector of Paisley in Scotland, are of interest: 17,576 tons of refuse, or an average of 50 tons per working day, was collected and dealt with in 1913, and 3177 tons of clinker and 243 tons of mortar were sold in that year.

The revenue of the destructor plant from the sale of the residual products was as follows:

Clinker .....	\$1,120
Mortar .....	240
Old tins and scrap metal .....	546
Waste paper .....	76
Old bottles .....	20
Total .....	\$2,006

In view of these figures, and of the fact that the relations between the sanitary and electrical departments at Huddersfield and Rotterdam are most harmonious, it would seem probable that a considerable increase will occur, during or after the war, in the number of destructor plants for towns' refuse equipped with the most efficient types of furnace, and that these plants will all derive considerable revenue from the sale of their surplus steam to the neighboring works and from the disposal of their clinker and other by-products to those who can make good use of these commodities in their operations.

If the experience of Huddersfield and of Paisley is a safe guide, an aggregate revenue of between \$11,800 and \$14,400 can be obtained in all large cities from these two sources alone, and a considerable portion of the cost of destroying the refuse can thus be met.

Colwyn Bay, via Liverpool,  
North Wales.

## Personal

Prof. Wilder D. Bancroft was in New York on Thursday, Feb. 8 and delivered a lecture in the evening at Columbia University on "Contact Catalysis" under the auspices of Gamma chapter, Phi Lambda Upsilon, and as a part of the extension teaching course.

Dr. V. A. Coulter has been appointed assistant professor of chemistry at the University of North Carolina.

Mr. Samuel H. Dolbear, consulting mining engineer, has removed his offices to 1411-12-15 Merchants' National Bank Building, San Francisco.

Mr. Gustav Drobegg has resigned his position with the Beckers Aniline & Chemical Co., Inc., to accept the superintendency of plant D of the Butterworth-Judson Corporation, at Newark, N. J.

General Geo. W. Goethals has opened consulting offices in the Wall Street Exchange Building, 43 Exchange Place, New York. A number of experienced specialists are associated with him, and a general consulting practice in civil, electrical, mechanical and hydraulic engineering will be engaged in.

Mr. Walter Kidde, engineer-constructor, of 140 Cedar Street, New York, has incorporated his organization under the title of Walter Kidde & Co., Inc. The business was established in 1900 and has grown steadily. One of their most recent commissions is the plant of the American Hard Rubber Co., at Akron, Ohio. Mr. Kidde's chief associates, who have been his partners on the "Carnegie Plan," will now become directors and stockholders of the new corporation, the officers of which are: Walter Kidde, president; B. G. Worth, vice-president; I. R. Lewis, secretary and treasurer. These are all members of the board of directors, which also includes Henry Lang, who is vice-president of the Ingersoll-Rand Company, and E. S. Boyer, who is associated with the American Hard Rubber Company. Mr. Kidde's other principal associates, who comprise the engineering board of the corporation, are: A. B. Miller, Walter S. Wainright, M. I. Buttfeld and E. Schwarz. The chief draftsman is Thorlief Fliflet.

Mr. A. R. Levin has become associated with the Supplee-Biddle Hardware Company as manager of the New York office, 30 Church Street.

Mr. O. C. Martin, who has been with the Nichols Copper Co., Laurel Hill, Long Island, for several years, has been made works manager of the company.

Mr. John G. Mason has become associated with Ralph L. Fuller & Co., Inc., 20 Rector Street, New York, as manager of their chemical and drug division.

Mr. J. Henry Rickard has recently returned to England from the United States.

Dr. A. H. Ney announces that he is in no way now connected with any consulting business or laboratory, firm of chemical engineers or manufacturers of chemical apparatus, and that any such connections which have existed, have been discontinued. His entire time is taken up with work in connection with the development of the chemical products and dye manufacturing plant of the Sherwin-Williams Company, of which he is the head, and he is not willing to consider propositions for his services in any form whatsoever, matters pertaining to national defense or economical development of the national resources alone excepted.

Mr. Paul Noble, recently with the Bayer Company, has become sales manager of the American Aniline Products, Inc., whose office has been removed from

15 East 12th Street, to 120 Hudson Street, New York City.

Professor E. F. Northrup, research physicist of Princeton University, has been awarded the Elliott Cresson medal by the Franklin Institute "in recognition of his electrical inventions and high temperature investigations."

Mr. Robert Carl Schroth, Jr., president of the Laboratory Supply Co., of Columbus, Ohio, and Mr. C. D. Fraunfelder, president of the Ohio Pottery Co., of Zanesville, Ohio, were in New York and attended the meeting on "Chemical and Electrical Porcelain" held at the Chemists' Club on Friday, Feb. 9. Both report great activity in their line.

Mr. A. M. Webb, assistant manager of the Chemical Construction Company, was married on January 11, to Miss Jean Harper, at Charlotte.

Professor W. M. Weigel has resigned as professor of mining at Pennsylvania State College, to become superintendent of the concentrating mills of the International Molybdenum of Renfrew, Ont.

### Book Reviews

**Pipe and the Public Welfare.** By R. C. McWane. 165 pages, illustrated. Price, \$1.00. New York: The Stirling Press.

This little volume was written for the purpose of showing the advisability of using cast-iron pipe for conduits and contains much that is of historical and practical interest. The author first discusses the history of the use of pipe in an interesting manner, beginning with the crude clay pipe of early Babylonian days, 4000 B. C. The history covers the introduction and development of iron and wood pipe, accounts of the water-supply systems of the large cities, and the use of iron pipe for gas mains. He then gives a review of materials and methods used in making metal pipe, which is followed by a long discussion on metal pipe deterioration, the latter being mostly in the nature of practical evidence of the rust-resisting qualities of cast iron. The last chapter is devoted to a discussion of the use of wood pipe. Concrete and clay pipe are purposely omitted, as these are not used to any extent in pressure conduits. Many interesting illustrations are included in the book.

**Selling Your Services.** By the Sales Service Company. Price, \$1. New York: The Sales Service Company, 627 Madison Avenue.

It is a little difficult at first for a technical man to think of his services or ability as a distinct commodity which can be marketed according to accepted principles of salesmanship. Yet here is a delightful little book which says that this is true and shows how to apply these principles to the obtaining of a position by a technical man. It is written in a convincing manner with many typical letters, and entirely from the salesman's viewpoint on accepted business principles.

**A Text Book of Inorganic Chemistry.**—Vol. VIII: The Halogens. By G. Martin and E. A. Dancaster. Crown octavo (15 x 22 cm.). 337 pages, 30 illustrations. Price, \$3. London: Charles Griffin & Company, Limited. Philadelphia: J. B. Lippincott Company.

Although numbered VIII in the series, it is the second to be issued. The other volumes are in preparation or in press. Both the editor of the series, Dr. J. Newton Friend, and the immediate authors of this volume emphasize the idea that the work is not intended to be exhaustive, that technical details are briefly touched

upon, and that most attention is given to the chemical and physical properties of the elements concerned and their compounds. The consequence of applying these principles is that the work is neither one thing nor another, neither an inspiring text-book nor a complete hand-book, but it has a certain value as a reference book if completeness is not expected, and from this viewpoint the present volume on halogens is not without merit.

**The Mineral Industry: 1915.** Edited by G. A. Roush. Octavo (15 x 23 cm.), XX + 941 pages; price \$10. New York: McGraw-Hill Book Company, Inc.

Volume XXIV looks as important and portly as its noted predecessors, while its pages, on inspection, bear out the statement that in quality of content it is the equal of its ancestors. There is not much need to praise it further than this, except to remark that the gathering of foreign data and statistics for 1915 was necessarily a work of the greatest difficulty, and everyone consulting the work will be impressed with the success attending the editor's efforts under such unusual handicaps.

Many of the regular contributors are represented (they are too many to catalog in a review), but several new and familiar ones have been added to the galaxy, such as J. W. Beckman on "Borax and Potassium Salts," R. M. Keeney on "Radium, Uranium and Vanadium," E. H. Leslie on "Gold and Silver in Mexico," E. S. Moore on "Gold and Silver in Australia and India," Baliol Scott on "Tin," L. O. Snyder on "Gypsum in Oklahoma," M. W. Von Bernewitz on "Gold and Silver in the United States," and on "Flotation."

**Report on the Fertilizer Industry.** By the Federal Trade Commission. U. S. Government Printing Office, August, 1916.

This 290-page report, prepared mostly by E. O. Merchant, F. L. Hawes and W. W. Bays, is full of valuable information, systematically arranged and written with judgment and snap. The successive chapters deal with inorganic nitrates and ammoniates, organic ammoniates, phosphates, potash salts, and mixed fertilizers. The technical, the commercial and the financial aspects are all fairly considered and skillfully interwoven. It constitutes a valuable contribution to the literature of this important industry, useful alike to the scientist and the farmer, the technologist and the business man. But it is an open question whether the report has not gone too far in divulging what might be considered "trade secrets" in fairness to the commercial interests concerned.

## CURRENT MARKET REPORTS

### The Iron and Steel Market

Germany's announcement of unrestricted submarine warfare, made public on the first of the month, was followed by a sudden and sharp decrease in inquiry for steel products. The market had already become relatively quiet in this respect in the fore part of December, on account of the German peace proposal, the close approach of the holidays and the influence that business engagements had already been made very far ahead. During January there had been no sign of the increase in market activity that usually follows the holiday period.

No noticeable decrease in the filing of specifications against old contracts followed the German announcement, nor was there any relaxation in the pressure exerted by buyers for heavier deliveries of material against old contracts. Buyers required time to reach conclusions as to what effect upon steel conditions would



be exerted by the probable consequences of Germany's action. Their conduct showed clearly that it was purely a case of taking time to consider. They would not make new engagements and they would not relax their efforts to continue the business they were conducting.

What occurred was exactly what would be expected of the steel market. It is sensitive in its way, but it is not sensitive like the non-ferrous metal market in respect to exhibiting price fluctuations in accordance with current news.

Two influences of opposing character were to be weighed. If the promised submarine campaign were a success our iron and steel exports would be greatly reduced. They were largely to points in the prescribed area, and there was a possibility of exports to South America and Canada being curtailed. In January the export movement involved, directly and indirectly, about 20 per cent of the steel production. An increase of 10 per cent in the domestic supply might have quite an effect.

On the other hand preparations by the United States for war would result in the placing of some rush orders for steel, and the steel producers were all perfectly ready to give such orders all the precedence desired. As it would be the height of inefficiency in such circumstances to fill such orders at the expense of deliveries to those already engaged in fighting the possible enemy of the United States, it would be domestic deliveries that would be delayed in consequence of the new orders.

#### CURTAILED PRODUCTION

Transportation conditions as affecting the production and shipment of steel were showing signs of slight improvement when a blizzard on Sunday, Feb. 4, swept over the Central West, in a territory producing at least half the country's iron and steel. Instantly railroad traffic was greatly impeded. Car supplies greatly decreased and the dispatch of loaded cars suffered. The chief disturbance, however, was in the Connellsville coke region, coke shipments in the week following the blizzard being probably no more than one-half the requirements of the tributary blast furnaces. Pig iron production in the region affected was already 20 per cent or more below normal and promised to drop to 50 per cent or less. Steel interests which for weeks had been considering themselves fortunate in having reserves of scrap and pig iron to draw upon were confronted with the prospect that these reserves would run out long before transportation conditions became such as to permit normal production of pig iron.

The cold snap froze up the steel mills badly and for a few days production was greatly restricted in some departments from that cause alone. Nearly all natural gas was shut off from the mills still using it, being required for domestic consumers, who by common practice are served first. A tin plate plant in the heart of the West Virginia gas field was closed because the available gas had to be piped to Pittsburgh and elsewhere for domestic consumption.

#### PIG IRON

Trends noticeable in January have developed farther. There is a more complete absence of interest on the part of consumers in second half deliveries. This is proved conclusively by the existence of an unprecedented condition, that in practically all northern consuming territory southern foundry pig iron is available for second half delivery at prices many dollars a ton below prices the northern furnaces would regard as proper prices in case they were called upon to quote. Their inquiries are confined to early deliveries and early deliveries alone can they sell. To quote lower prices for forward de-

liveries would only embarrass them in making prompt sales. For illustration, the last important sales of valley pig iron for second half delivery, made some time ago, were at \$30, valley, for foundry or basic. Of late the furnaces have been obtaining \$33 to \$35 for small lots of prompt iron, which would be \$33.95 to \$34.95 delivered Pittsburgh, while for second half delivery Southern foundry iron is available at not over \$24, Birmingham, or \$28.55 delivered Pittsburgh. It is available for prompt shipment at the same price, but delivery would be very uncertain on account of the long haul. The normal pig iron market is one for forward deliveries and now there is no such market.

Export pig iron inquiry has diminished greatly. As the heavy export demand had a great deal to do with advancing and maintaining pig iron prices the new international conditions impose even more conservatism upon pig iron buyers than upon steel buyers.

#### STEEL

About February 1 a sale of 3000 tons of soft, open-hearth billets was made at \$65, f.o.b. maker's mill, Pittsburgh district. Transactions are few and far between and this one is probably representative of the market as far as there is a market.

Finished steel prices are firm all along the line. The advance in shapes and plates of \$3 a ton made by most of the mills in January has become general and prices for delivery at mill convenience may be quoted at 3c. for bars, 3.25c. for shapes and 3.75c. for plates. Large premiums rule on plates for earlier delivery, and small premiums on bars and shapes. Sheets and wire products are steady, with suspicions on the part of buyers that the producers are not sold up as fully as appeared a month ago to be the case. Tin plate is practically unobtainable, with \$8 bid in some instances. The mills are sold very fully to July 1 on tin plate and are altogether indisposed to consider the matter of prices for second half.

#### Non-Ferrous Metal Market

*Feb. 8.*—The disturbed international conditions have not caused very great changes at the present writing excepting in tin, which has gone higher in a somewhat excited market. Copper is slightly higher on heavier inquiry and antimony is scarce and higher. Spelter and lead are firm and practically unchanged.

*Copper.*—Sellers have been extremely shy for the greater part of the last two weeks, and little copper has been offered for early delivery. Electrolytic was offered at 33.50 on Jan. 29, at 33.00 on Feb. 1, and at 34.50 on Feb. 7. Lake has been running about 1 cent under this price. Exports in January were 23,511 tons. On Feb. 7 more inquiry was shown than previously and prices went up.

*Tin.*—As is to be expected from a market depending on foreign shipping, the tin market is unsettled and no one knows what will happen from one day to the next. There has been considerable activity in Banca tin at prices ranging from 44.50 on Jan. 30 to 51.00 cents on Feb. 7. The New York Metal Exchange gives deliveries for January at 7177 tons, of which 5200 tons were at Atlantic ports and 1977 tons at Pacific ports. This is 1200 tons above the record. Straits is quoted at 55.00 cents.

*Lead.*—On Jan. 29 the trust price was advanced to 8.00 cents. Independents have been asking from  $\frac{1}{2}$  to 1.00 cent above the trust for some time, and the raise was not unexpected. Lead is scarce and there has been practically no market on spot. At present independents are asking 8.50 to 9.00 cents, prompt delivery.

**Spelter.**—Ore prices were advanced on Jan. 29, and this was followed by an advance in the metal to 11.05 cents, New York. It was reported on Jan. 31 that Great Britain would not buy any more spelter here, but the report had little effect. Spelter is now quoted at 10.55, New York, for prompt shipment.

**Other Metals.**—Antimony has become very scarce and has jumped from 23.00 cents on Jan. 29 to 32.00 cents on Feb. 7. Aluminium is now quoted at 57.00 to 59.00 cents, magnesium is down to \$3.00, electrolytic nickel is 50.00 cents, cadmium \$1.50, quicksilver \$100.00, platinum \$100.00, cobalt \$1.50, and silver 77½¢.

### Chemical Market

Should the diplomatic break with Germany eventuate into open hostilities the chemical markets, particularly the coal tar division, will of course attract national attention. At the moment of writing sufficient time has not elapsed to effect material changes. Sellers, however, have adopted a very cautious attitude and the general tendency is to hold off pending developments.

**Benzol** has been subjected to considerable activity during the interval and the general asking rate of sellers has been on a higher level than when we last reported. One producer has been a disturbing influence, however, and some of their output was offered at a price considerably under the general market. However, only the most favored buyers could secure this material and it is believed that most of this product has now been sold. The largest selling interest has been accepting contracts over the three-year period with a guarantee against declines. It is estimated that the production of benzol at the present time is approximately 40,000,000 gal. yearly.

The **toluol** situation has become very interesting. In view of the probability of the American government shortly coming into the market for supplies of *T. N. T.* the general tendency is to hold supplies and there is a pronounced speculative interest present that is attempting to secure such supplies that may be available at attractive prices.

Again with **phenol**, the possibility of large orders for picric acid developing has once more created a firmer feeling in this market after a pronounced weak tendency for the past two weeks. Offerings from the middle West had resulted in the lowest prices that have prevailed for a long time and in consequence Eastern producers were compelled to meet this competition.

**Aniline oil**, however, has maintained an unusual degree of firmness during the interval and this situation was not in any way a reflection of political conditions. There are approximately 45 plants equipped with apparatus for the manufacture of aniline oil or its homologues in this country and the actual producers at the moment do not probably exceed 15. This is due to the unusually low prices that have been prevailing of late making it possible for only the most favorably situated from the raw material viewpoint to manufacture profitably. The production has decreased largely and a large number of export orders have reached this market of late resulting in an advance of more than 5 cents per lb. during the fortnight.

**Ortho and para toluidine** have become scarce with prices high as a result of the restricted production and the difficulty of separation. **Toluidine** mixture has been made in a fair way but unseparated has found but limited markets. A production of **tolidine** is announced by a prominent company, which has heretofore principally confined its operations to *alpha-naphthylamine*. **Commercial tolidine** base is held at \$3 for prompt delivery with producers not open for contracts at this time.

**H acid** (the *amidonaphthol disulphonic acid* 1:8:3:6) continues to attract a vast amount of attention. There is but little doubt that with the endless discussion of this product there will be a number of producers within the year. Already a production is announced for the near future by two entirely new factors who have not heretofore figured in this field.

**Heavy Chemicals.**—The possibility of restricted exports has generally tended to lower prices in this department of the industry. Products such as *copper sulphate*, which depend to a large degree on the export trade for their stability, slumped rather sharply. Weakness still prevails and general conditions are unsettled.

**Caustic soda** and **soda ash** have been working rather well in unison. The break with Germany resulted in a sharp decline in the former product but during the intervening few days there has been some quiet important buying and the general tendency at the moment of writing is to a recovery of lost values.

**Glycerine** has naturally been subject to much attention as a result of the international situation and an advance of several points has already been scored. The situation in this product is always more or less under stable control and a firmer market is likely. **Formaldehyde** has been scarce and considerable business has passed for domestic and export account.

Owing to the more liberal offerings of *cyanides* from abroad the situation has eased off considerably. The domestic situation is tight but as most material offered of late has been in speculative hands these interests have weakened in the face of liberal foreign offers principally from Japan.

In pharmaceuticals, *citric acid* and *citrates* have advanced sharply in face of the probability of more than an usual brisk spring demand. *Mercury* and *mercurials* have also advanced as has *cream of tartar*.

Interesting developments are expected in the market during the next few weeks and to-day's prices have but little meaning or value to-morrow. The crisis that has developed finds the chemical trade generally in better shape to assist the national government than ever before and already a number of the leading chemical manufacturers have offered the facilities of their plants to Washington.

### General Chemicals

WHOLESALE PRICES IN NEW YORK MARKET FEBRUARY 7, 1917

Acetone, Drums .....	lb.	23	—	23 1/2
Acid, acetic, 28 per cent. ....	100 lb.	3.25	—	3.50
Acetic, 56 per cent. ....	100 lb.	6.75	—	7.00
Acetic, glacial, 99 1/2 per cent, carboys. ....	lb.	.22	—	.23
Boric, crystals .....	lb.	.11 1/2	—	.11 3/4
Citric, crystals .....	lb.	.72	—	.80
Hydrochloric, commercial, 18 deg. ....	lb.	.01 1/4	—	.01 3/4
Hydrochloric, 20 deg. ....	lb.	.01 1/4	—	.01 1/2
Hydrochloric, C. P., conc., 22 deg. ....	lb.	.01 3/4	—	.01 1/2
Hydrofluoric, 30 per cent. in barrels. ....	lb.	.04 1/2	—	.05
Lactic, 44 per cent. ....	lb.	.11 1/2	—	.12
Lactic, 22 per cent. ....	lb.	.04 1/2	—	.05
Nitric, 36 deg. ....	lb.	.04 1/4	—	.04 1/2
Nitric, 42 deg. ....	lb.	.05	—	.05 1/4
Oxalic, crystals .....	lb.	.46	—	.48
Phosphoric, 85 per cent. ....	lb.	.30	—	.32
Picric .....	lb.	.70	—	.75
Pyrogallol, resublimed .....	lb.	3.25	—	3.30
Sulphuric, 66 deg., Brimstone. ....	ton	24.00	—	25.00
Sulphuric, 60 deg., Brimstone. ....	ton	20.00	—	21.00
Sulphuric, oleum (Fuming), tank cars. ....	ton	34.00	—	35.00
Tannic, tech. ....	lb.	.45	—	.50
Tartaric, crystals .....	lb.	.72	—	.85
Alcohol, grain, 188 proof. ....	gal.	2.72	—	2.74
Alcohol, Wood, 95 per cent. ....	gal.	1.00	—	1.02
Alcohol, Denatured, 180 proof. ....	gal.	.66	—	.67
Alum, ammonia lump. ....	lb.	.04 1/8	—	.04 1/2
Alum, chrome (ammonium). ....	lb.	.21	—	.23
Alum, chrome (potassium). ....	lb.	.30	—	.34
Alum, potash lump. ....	lb.	.05 1/2	—	.05 3/4
Aluminum Sulphate, technical. ....	lb.	.01 1/8	—	.02
Aluminum Sulphate, iron free. ....	lb.	.03	—	.03 1/8
Ammonia aqua, 26 deg., carboys. ....	lb.	.05 1/2	—	.06
Ammonia anhydrous .....	lb.	...	—	...
Ammonium, carbonate .....	lb.	.12 1/2	—	.13
Ammonium, nitrate .....	lb.	.14	—	.14 1/4
Ammonium sulphate, domestic. ....	lb.	.04	—	.04 1/4
Amyl acetate .....	gal.	4.00	—	4.25
Arsenic, white .....	lb.	.10 1/2	—	.11
Arsenic, red .....	lb.	.25	—	.30
Barium chloride .....	ton	92.00	—	95.00



Barium sulphate (Blanc Fixe) powder.....lb.	.04 1/4	.04 1/2
Barium nitrate.....lb.	.12	.12 1/2
Barium peroxide, basis 70 per cent.....lb.	.28	.30
Bleaching powder, 35 per cent (large drums).....lb.	.03 3/4	.04 1/4
Borax, crystals, sacks.....lb.	.08 1/4	.08 1/4
Brimstone, crude.....ton	28.50	29.00
Bromine, technical.....lb.	1.30	1.35
Calcium acetate, crude.....lb.	.03 1/4	.03 1/2
Calcium carbide.....ton	85.00	90.00
Calcium chloride, 70-75 per cent, fused, lump.....ton	23.50	25.00
Calcium peroxide.....lb.	1.70	1.75
Calcium sulphate.....lb.	.01	.01 1/2
Calcium phosphate.....lb.	.30	.31
Carbon bisulphide.....lb.	.04 1/2	.04 3/4
Carbon tetrachloride, drums.....lb.	.15 1/2	.16
Caustic potash, 82-92 per cent.....lb.	.85	.88
Caustic soda, 76 per cent.....lb.	.04	.04 1/4
Chlorine, liquid.....lb.	.15	.16
Copperas.....100 lb.	1.15	1.25
Copper carbonate.....lb.	.37	.39
Copper cyanide.....lb.	.70	..
Copper sulphate, 99 per cent, large crystals.....lb.	.10	.11
Cream of tartar, crystals.....lb.	.42	.42 1/2
Epsom salt, bags.....100 lb.	.01 1/4	.02
Formaldehyde, 40 per cent.....lb.	.12 1/2	.12 1/2
Glauber's salt.....100 lb.	.65	.75
Glycerine, bulk, C. P.....lb.	.54 1/2	.55
Iodine, resublimed.....lb.	3.50	3.55
Iron oxide.....lb.	.02	.08
Lead acetate, white crystals.....lb.	.13 3/4	.14 1/2
Lead arsenate.....lb.	.09	.09 1/4
Lead nitrate.....lb.	.16 1/4	.16 1/2
Litharge, American.....lb.	.09	.09 1/4
Lithium carbonate.....lb.	1.02	1.03
Manganese dioxide.....lb.	.65	.70
Magnesium carbonate, tech.....lb.	.13	.13 1/4
Nickel salt, single.....lb.	.14	..
Nickel salt, double.....lb.	.11	..
Phosphorus, red.....lb.	1.08	1.15
Phosphorus, yellow.....lb.	.60	.65
Potassium bichromate.....lb.	.37 1/2	.38
Potassium bromide, granular.....lb.	1.20	1.25
Potassium carbonate, calcined, 80-85 per cent.....lb.	.30	.32
Potassium chlorate, crystals.....lb.	.64	.65
Potassium cyanide, 98-99 per cent.....lb.	2.35	2.40
Potassium iodide.....lb.	2.90	2.92
Potassium muriate, 80-85 per cent basis of 80 per cent.....ton	400.00	425.00
Potassium nitrate.....lb.	.30	.32
Potassium permanganate.....lb.	3.75	4.00
Potassium prussiate, red.....lb.	2.50	2.75
Potassium prussiate, yellow.....lb.	.90	.92
Potassium sulphate, 90-95 p.c. basis 90 p.c. ton	300.00	325.00
Rochelle salts.....lb.	.34	.34 1/2
Sal ammoniac, gray gran.....lb.	.09	.09 1/2
Sal ammoniac, white gran.....lb.	.17	.17 1/2
Sal soda.....100 lb.	1.10	1.15
Salt cake.....100 lb.	.70	.75
Silver cyanide, based on silver market.....oz.	.72	..
Silver nitrate.....oz.	.47 1/2	.49 1/2
Soda ash, 58 per cent, light, flat.....100 lb.	2.75	2.85
Soda ash, 58 per cent, dense, flat.....100 lb.	3.50	3.75
Sodium acetate.....lb.	.08	.08 1/2
Sodium benzoate.....lb.	7.50	8.00
Sodium bicarbonate, domestic.....100 lb.	1.90	2.00
Sodium bicarbonate, English.....lb.	.03	.03 1/2
Sodium bichromate.....lb.	.14 1/2	.15
Sodium bisulphite, powd.....lb.	.04 3/4	.05
Sodium chlorate.....lb.	.25	.26
Sodium cyanide.....lb.	1.25	1.30
Sodium fluoride, commercial.....lb.	.12	.13
Sodium hyposulphite.....lb.	.01 5/8	.01 3/4
Sodium nitrate, refined.....100 lb.	3.40	3.45
Sodium nitrite.....lb.	.11 1/2	.11 3/4
Sodium peroxide.....lb.	.90	1.10
Sodium phosphate (tri.).....lb.	.04 1/2	.05
Sodium prussiate, yellow.....lb.	.33	.34
Sodium silicate, liquid, 40-50 deg.....100 lb.	1.50	1.60
Sodium sulphide, 50 per cent crystals.....lb.	.03	.03 1/2
Strontium nitrate.....lb.	.30	.32
Sulphur chloride, drums.....lb.	.09 1/2	.10
Sulphur dioxide, cylinders.....lb.	.11 1/2	..
Sulphur, flowers, sublimed.....100 lb.	2.30	2.40
Sulphur, roll.....100 lb.	1.90	2.00
Sulphur, crude.....ton	36.00	38.00
Tin bichloride, 50 deg.....lb.	.14	.14 1/2
Tin oxide.....lb.	.47	.49
Zinc carbonate.....lb.	.26	.27
Zinc chloride.....lb.	.13	.13 1/4
Zinc cyanide.....lb.	.50	..
Zinc dust.....lb.	.20	.24
Zinc oxide, American process XX.....lb.	.11 1/4	.11 3/4
Zinc sulphate.....lb.	.06	.06 1/2

## Coal Tar Products (Crude)

Benzol, pure, water white.....gal.	.55	.57
Benzol, 90 per cent.....gal.	.55	.58
Toluol, pure, water white.....gal.	1.75	1.80
Xylol, pure, water white.....gal.	1.00	1.15
Solvent naphtha, water white.....gal.	.21	.22
Solvent naphtha, crude heavy.....gal.	.13	.15
Creosote oil, 25 per cent.....gal.	.25	.26
Dip oil, 20 per cent.....gal.	.22	.24
Pitch, various grades.....ton	7.50	20.00
Carbolic acid, crude, 95-97 per cent.....lb.	.85	.95
Carbolic acid, crude, 50 per cent.....lb.	.60	.65
Carbolic acid, crude, 25 per cent.....lb.	.25	.27
Cresol U. S. P.....lb.	.18	.22

## Intermediates, Etc.

Alpha naphthylamine.....lb.	1.00	1.25
Aniline oil.....lb.	.25	.28
Aniline salts.....lb.	.27	.30
Anthracene, 80 per cent.....lb.	.104	..
Benzaldehyde.....lb.	4.25	4.50
Benzidine, base.....lb.	1.60	1.75

Benzoic acid.....lb.	8.50	9.00
Beta naphthol, sublimed.....lb.	.85	.90
Dichlor benzol.....lb.	.48	.55
Dimethylaniline.....lb.	.55	.60
Diphenylamine.....lb.	.80	.85
H-acid.....lb.	Nominal at 2.50	..
Metaphenylenediamine.....lb.	1.60	1.75
Monochlorbenzol.....lb.	.32	.33
Naphthalene, flake.....lb.	.09	.10
Naphthionic acid, crude.....lb.	1.75	2.00
Ortho-toluidine.....lb.	..	..
Ortho-toluidine.....lb.	1.25	1.50
Para-amidophenol, base.....lb.	4.25	4.50
Para-amidophenol, sulphate.....lb.	4.50	4.75
Para-amidophenol, hydrochloride.....lb.	5.50	6.00
Paranitraniline.....lb.	1.35	1.45
Paraphenylenediamine.....lb.	3.50	3.75
Para toluidine.....lb.	1.50	1.75
Phenol, U. S. P.....lb.	.47	.50
Resorcin, technical.....lb.	9.00	..
Resorcin, pure.....lb.	16.50	17.00
Salicylic acid.....lb.	.85	.90
Salol.....lb.	1.50	1.55
Sulphanilic acid.....lb.	.35	.36
Tolidine, base.....lb.	3.00	..
Toluidine-mixture.....lb.	.65	.90

## Petroleum Oils

## CRUDE (AT THE WELLS)

Pennsylvania.....bbl.	3.05	..
Corning, Ohio.....bbl.	2.38	..
Somerset, Ky.....bbl.	2.18	..
Wooster, Ohio.....bbl.	2.05	..
Indiana.....bbl.	1.73	..
Illinois.....bbl.	1.90	..
Oklahoma and Kansas, except Healdton.....bbl.	1.70	..
Healdton, 32 deg. and above.....bbl.	.90	..
Caddo, La., light.....bbl.	1.70	..
Corsicana, Tex., light.....bbl.	1.70	..
California.....bbl.	.73	.82

## LUBRICANTS

Black, reduced, 29 gravity, 25-30 cold test.....gal.	13 1/2	14
Cylinder, light.....gal.	.21	.26
Cylinder, dark.....gal.	.18	.19
Extra cold test.....gal.	.26	.31
Paraffine, high viscosity.....gal.	29 1/2	30
Paraffine, 0.903 spec. gr.....gal.	21 1/2	22
Paraffine, 0.865 spec. gr.....gal.	18 1/2	19

## Flotation Oils

Pine oil, steam distilled.....gal.	.59	..
Pine oil, destructively distilled.....gal.	.47	..
Pine-tar oil.....gal.	.19	..
Pine-tar oil, double refined.....gal.	.30	..
Pine oil, light.....gal.	.37	..
Pine oil, heavy.....gal.	.26	..
Pine tar, thin.....gal.	.18	..
Turpentine, crude.....gal.	.38	..
Hardwood oil, f.o.b. Michigan.....gal.	.16	..
Creosote, coal tar, neutral.....gal.	.15	..
Creosote, coal tar, acid.....gal.	.21	..
Coal tar, thin.....gal.	.12	..

## Vegetable and Other Oils

China wood oil.....gal.	.13 1/2	..
Cottonseed oil, crude.....gal.	.80	.82
Linseed oil, raw, cars.....gal.	.94	..
Peanut oil, crude.....gal.	.88	.90
Rosin, 280 lb.....bbl.	6.30	..
Rosin oil, first run.....gal.	.38	..
Rosin oil, fourth run.....gal.	.70	..
Soya bean oil, Manchuria.....gal.	.12	..
Sperm oil, bleached winter, 38 deg.....gal.	.12	.12 1/4
Turpentine, spirits.....gal.	.52 1/2	..

## Miscellaneous Materials

Barytes, floated, white, domestic.....ton	25.00	35.00
Beeswax, white pure.....lb.	.48	.50
Carnauba wax, highest grade.....lb.	.50	.51
Chalk, light, precipitated, English.....lb.	.03	.06
Feldspar.....ton	8.00	12.00
Fuller's earth, powdered.....100 lb.	.80	1.05
Red lead, dry, carloads.....lb.	.09 3/4	..
Soapstone.....ton	10.00	12.50
Talc, American, white.....ton	10.00	13.00
White lead, dry.....lb.	.08 3/4	..

## Refractories, Etc.

## (F.O.B. WORKS)

Chrome brick.....net ton	120.00	130.00
Chrome cement, Grecian.....net ton	80.00	90.00
Clay brick, first quality fireclay.....per 1000	40.00	50.00
Magnesite, Grecian, dead burned.....net ton	85.00	90.00
Magnesia brick, Grecian, 9x4 1/2 x 2 1/2.....net ton	135.00	140.00
Silica brick.....per 1000	40.00	45.00

## Ferroalloys

Ferro-carbon-titanium, carloads.....lb.	.08	..
Ferromanganese, carloads.....lb.	.19	..
Ferromanganese, domestic, delivered, contract.....ton	165.00	..
Ferromanganese, English, contract.....ton	175.00	..
Ferromolybdenum.....lb.	4.00	..
Ferrosilicon, 50 p.c., carloads, del. Pittsburgh.....ton	140.00	200.00
Ferrosilicon, 50 p.c., contract, del. Pittsburgh.....ton	100.00	..
Ferrotungsten, 75-85 p.c., f.o.b. Pittsburgh.....lb.	2.10	2.15
Ferrovanadium, f.o.b. works.....lb.	2.75	3.00

# INDUSTRIAL

## Financial, Construction and Manufacturers' News

### Financial

Atlas Refining Company, Delaware, has been incorporated with a capital of \$150,000 to produce petroleum and its products. The incorporators are C. W. Kirkland, Mukogee, Okla.; P. W. Murray, Bridgeport, Conn.; J. P. Given, New York; E. Walter Mitchell, Brooklyn, and Count Thomas Devassay, Budapest, Austria Hungary.

The Auto Products Company, Nashville, Tenn., has been incorporated with a capital of \$2,000. Incorporators are A. E. Potter, A. Odell, F. M. Kelly, J. C. Ransom, P. C. Bowers, S. C. Ewing, F. H. Turner, H. G. Tucker and W. H. Ewing. The company expects to make benzoic acid and other chemicals.

Canton Metal Products Company, Canton, Ohio, has been incorporated with a capital of \$1,000,000. Incorporated by H. G. Bow.

The Carbonate Copper Company, San Antonio, Tex., has been incorporated with a capital of \$10,000. The incorporators are E. L. Porch, W. Schertz and W. F. Miller.

The Carlo-Nitrate Company, Pittsburgh, Pa., has increased its capital from \$5,000,000 to \$7,500,000.

The Cincinnati Iron and Steel Company has increased its capital stock from \$400,000 to \$800,000.

The Clover Foundry Company, Muskegon, Mich., has been incorporated with \$50,000.

Coeur-d'Alene Copper Mining Company, Spokane, Wash., has been incorporated with a capital of \$25,000. The incorporators are E. A. Laing, L. Harvey, M. A. Schneider.

Comstedt & Company, Inc., New York, has been incorporated with a capital of \$100,000. The incorporators are G. L. R. Munson, H. Waddington, J. T. A. Comstedt, 120 Broadway. The company is authorized to conduct business in iron, steel, and metal work.

Copper Products Company, Augusta, Me., has been incorporated with a capital of \$250,000 to carry on a general business of foundry, machine shop, manufacturing, depositing metals on other metals, and other business. The incorporators are S. S. Stevens, Manchester, Mass.; H. P. Sweetser, Portland; E. C. Ramsdell, Boston; P. E. Coyle, A. P. Brewer, Portland.

Driggs Mfg. Corp., New York, has been incorporated with a capital of \$300,000 to engage in factory supplies, ammunition, explosives, marine craft, aeroplanes. The incorporators are W. Zane, W. Abramson, L. L. Driggs, Jr., 120 Broadway.

The Durham Iron Works, Inc., Durham, Va., has been incorporated with a capital of \$25,000 for general foundry and machine shops. The incorporators are W. R. Kreker, W. F. Muelerschoen, and others.

The E. I. du Pont de Nemours Powder Company is said to have offered \$1,000,000 for the plants and business of Harrison Bros. & Company, Inc. The Harrison Bros. Company has large plants at Philadelphia and Paulsboro, N. J., and manufactures paints, pigments, and chemicals. It is also the owner of pyrite mines in Virginia. Russell S. Hubbard is president and general manager of the Harrison Bros. Company.

Eastern Chemical Company, Wilmington, Del., has been incorporated with a capital of \$200,000.

The Electric Chemical Company, Salisbury, N. C., has been incorporated with a capital of \$10,000. The incorporators are M. A. Hodgkin, C. L. Jurkley, and others.

M. M. Elish & Company, Inc., Manhattan, has been incorporated with a capital of \$100,000 to manufacture paper. The incorporators are M. M. Elish, 620 E. 26th Street, Brooklyn; L. H. Biglow, Jr., South Orange, N. J.; H. L. Gutterson, Beechmont Park, New Rochelle.

The Excelsior Chemical Company, Youngstown, Ohio, has been incorporated with a capital of \$15,000. The incorporators are J. W. Blackburn, J. P. Wilson, C. W. Osborne, F. J. Helm, R. B. Wilson.

J. B. Gathright Land Company, Louisville, Ky., has been incorporated with a capital of \$25,000 to deal in oil, gas and other mineral properties. Incorporators,

W. H. Field, Matt O'Doherty, J. S. Clark, H. Harrison.

The General Chemical Company doubled its net profits in 1916. The amount for the year ending Dec. 31, 1916, was \$12,481,826, compared with \$6,153,796 for the year ending Dec. 31, 1915.

Great American Refining Company, Indianapolis, Ind., has been incorporated in Delaware with a capital of \$10,000,000 to produce oil and its products. The incorporators are Jos. O. More, Orpheus L. Teague, L. E. Replogle, all of Indianapolis.

Grasselli Chemical Company earnings were reported at \$9,935,000 for 1916, or slightly better than 70 per cent of the present common stock issue of \$13,913,000, after deducting the preferred stock dividend requirements. In 1915 the company earned \$4,859,000.

The Harley Company, Springfield, Mass., has been incorporated to manufacture machinery and tools with a capital of \$1,200,000. The incorporators are L. J. Harley, L. J. Harley, Jr., Chas. H. Beckwith, all of Springfield.

Holland Aniline Dye Company, Holland, Mich., has been incorporated with a capital of \$250,000. The \$200,000 stock recently placed on the market has been subscribed.

The Holland Manufacturing Company, Cleveland, Ohio, has been incorporated with a capital of \$10,000 to deal in metal products. Incorporators, Jos. Hollander, Lena Hollander, H. Schanberg, J. Holstein and J. Hudik.

Industrial Paint Works, Inc., Passaic, N. J., has been incorporated with a capital of \$100,000 to manufacture paints and painters' supplies. The incorporators are R. Huff, J. P. Salisbury, F. A. Katterson.

International Peroxide Company, Brooklyn, N. Y., has been incorporated with a capital of \$5,000 to deal in and manufacture chemicals. Incorporators, J. R. Levine, I. Heitler, W. Wisch, 45 Malta Street, Brooklyn.

The Iowa Portland Cement Company, Iowa, has been purchased by the Lehigh Portland Cement Company, a Pennsylvania corporation operating cement plants in Pennsylvania, Virginia, Indiana, Washington, Illinois and Mason City, Ia.

The new owners will continue the policy of increasing the efficiency of the Iowa plant. The Iowa Portland Cement Company, a 2 1/2 million concern with general offices in the Commerce Building, Kansas City, expended \$300,000 in modernizing the plant last year and a similar outlay is to be made this year.

H. Struckmann, J. J. Heim, R. A. Long, J. W. Perry and A. D. Bayles are among the Kansas City stockholders of the Iowa Company. The largest stockholder has been the National Bank of Commerce of St. Louis.

The Iowa Pure Iron Company, De Moines, Iowa, has been incorporated with a capital of \$100,000 to deal and manufacture sheet iron metal products. Incorporators are H. L. Helsing, E. Bern, A. G. Helsing.

Kennecott-Barrett Copper Company, Seattle, Wash., has been incorporated with a capital of \$1,500,000. The incorporators are Geo. Miller, T. C. Brownlee, W. A. Gaines and Geo. A. Lee.

Kennecott Extension Copper Company, Wilmington, Del., has been incorporated with a capital of \$5,000,000 to prepare all kinds of metals and minerals for market. The incorporators are M. L. Rogers, L. A. Irwin, H. W. Davis, all of Wilmington.

Keystone Mfg. Company, New Kensington, Pa., has been incorporated with a capital of \$30,000 to manufacture various articles of iron, steel, etc. The incorporators are J. P. Posahon, M. G. Penney, C. J. Lange.

Lukens Steel Company, Coatesville, Pa., has been incorporated with a capital of \$10,000. Gustave T. Schnatz, Philadelphia, treasurer.

Mechanical Window Glass Company, Springdale, Pa., has been incorporated with a capital of \$5,000,000 to manufacture window and plate glass. The incorporators are Geo. C. Debay, Springdale; Warren D.

Gumpp; Arnold, Pa., and H. L. Schenck, of Pittsburgh.

Michel-Bilodeau Chemical Company, San Francisco, Calif., has been incorporated with a capital of \$50,000 to deal in drugs, chemicals and toilet articles. The incorporators are H. Michel, L. Bilodeau, F. B. Canas-Marquis.

The National Chemical Company, Ltd., with offices in the People's Gas Bldg., Chicago, has recently been incorporated for \$1,250,000 in S. Dakota and Illinois. The company will manufacture potash and by-products from kelp. It has been granted an exclusive concession by the Canadian Government on kelp beds at Pacofi, Queen Charlotte Island, B. C., which are estimated to produce 1,000,000 tons of kelp annually. The company's holdings are also in an excellent fishing district and it is expected to make the returns from fish fertilizer an important part of the company's income. Mr. M. V. Coone is president and general manager; Mr. Geo. A. Schwabland, who has had much experience in the kelp industry, is the company's chemical engineer.

New Jersey Dyestuffs Corp., Jersey City, N. J., has been incorporated with a capital of \$25,000 to deal in dyestuffs and chemicals of all kinds. The incorporators are R. Schroeder, N. Marcus, R. Rieser, of Hoboken.

Nicholson Furnace Mfg. Company has been incorporated in Delaware with a capital of \$200,000 to manufacture furnaces, stoves, etc. The incorporators are J. J. Fry, L. T. Hopkins, and George H. Harman, all of New York.

Nome Sunset Mines, Inc., Nome, Alaska, has been incorporated in Delaware with a capital of \$2,500,000 to engage in mining of all kinds. The incorporators are J. Tobin, A. F. McIntosh, Jas. Halpin.

The North & South American Oil Company, Wilmington, Del., has been incorporated with a capital of \$2,000,000 to deal in oil lands and mining properties.

Old Dominion Chemical Company, Inc., Yorktown, Va., has been incorporated with a capital of \$275,000 to manufacture chemicals. The incorporators are A. D. Christian and E. S. Bolen, both of Richmond.

The Penant Produce Company, Cleveland, Ohio, has been incorporated with a capital of \$1,000,000 to manufacture and sell metal products of all kinds. Incorporators are R. D. Stevenson, G. R. Stevenson, L. B. Foote, W. A. Thompson, A. J. Hudson, all of Cleveland.

Pole Star Copper Company, Salt Lake City, Utah, has been incorporated with a capital of \$1,500,000. The business of the company will be mining, milling, smelting, etc. The incorporators are H. E. Latter, N. P. Coffin and H. T. Farrow.

Renania Chemical Works, Inc., New York, has been incorporated with a capital of \$15,000 to manufacture and deal in chemicals and medicines. The incorporators are J. H. Hutton, G. A. Wortelman, J. V. Bendus, 17 South Street.

The Reward Chemical Company, Toledo, Ohio, has been incorporated with a capital of \$10,000. The incorporators are J. L. Greenbaum, A. A. Greenbaum, Christ. Hopp, L. J. Mechler, and Sadie N. Joyce.

Roseburgh Chemical Corp., Syracuse, N. Y., has been incorporated with a capital of \$50,000 to manufacture chemicals. The incorporators are J. E. Porter, T. Hiseock, R. M. Roseburgh.

Royal Dye Works, Inc., New York, has been incorporated with a capital of \$10,000 to deal in dyes and chemicals. The incorporators are F. P. Kamholz, L. Scorsone, W. Koenig, 25 Echo Road, New Rochelle.

Serrenita Mining Company has been incorporated in Delaware with a capital of \$100,000 to explore mines for ores, minerals, etc. The incorporators are V. C. Bogardus, H. H. Walter, M. Friedberg, all of New York.

Shure White Chemical Company, Mayfield, Ky., has been incorporated with a capital of \$5,000 to manufacture chemicals. The incorporators are L. Evans, W. W. Evans and N. E. Thomas.

Stockholders of the Solvay Process Company will vote on Feb. 27 to increase the capital stock from \$10,000,000 to \$20,000,000. Of the increase \$2,000,000 is to be offered to the share owners for subscription on the basis of 20 per cent of their holdings, as of March 1.

Springfield Wall Paper & Paint Company, Springfield, Mass., has been incorporated with a capital of \$50,000. The incorporators are J. Howard Jones, Geo. W. Steele and W. H. McCarthy.

The Standard Gas Power Company, 17 Battery Place, New York, is undergoing



a reorganization which will take several months. Mr. Akerlund, formerly chief engineer, has formed a company to be known as Akerlund & Semmes, 17 Battery Place, New York, to carry on combustion engineering business. It is possible that this company may be included in the reorganization of the Standard Gas Power Company.

Standard Oil Gas Burner Company, Wilmington, Del., has been incorporated with a capital of \$30,000 for manufacture and sale of oil gas burners.

Steffanson & Company, Inc., New York, has been incorporated with a capital of \$100,000 to manufacture paper. Incorporators, C. Engel, R. N. Chambers, J. French, 59 Wall Street.

The Stockholders Mining Company, Portland, Me., has been incorporated with a capital of \$2,000,000 to engage in mining, quarrying, smelting, refining, copper, gold, silver and all kinds of ores, etc.

B. T. Tuttle and W. W. Gregory, of Roscoe, N. Y., have formed a partnership for the manufacture of dyes. A factory will be erected in the spring.

The Union Oil Company, of California, in its financial report for the year ending Dec. 31, 1916, shows the company to have had a most successful year. Net profits are given as \$7,200,000, or \$4,380,000 greater than 1915.

United Chemical Company, Pittsburgh, Pa., has been incorporated with a capital of \$5,000. Incorporator, Jas. D. White.

United States Chemical Company, New Haven, Conn., has been incorporated with a capital of \$50,000 to manufacture and deal in chemicals. The incorporators are J. H. Sheehan, D. M. Freeman, J. J. Dunlap, all of New Haven.

Utah Mineral Paint Company, Salt Lake City, Utah, has been incorporated with a capital of \$50,000. The incorporators are C. H. Barton, David Jensen and Geo. A. Barry. Headquarters will be in Ogden.

The Val Blatz Paint & Varnish Company, Louisville, Ky., has changed its name to the Blatz Company and increased its capital from \$10,000 to \$30,000.

Virginia-Tennessee Iron Corp., Clifton Forge, Va., has been incorporated with a capital of \$100,000. Incorporators, C. F. Sentz, A. C. Ford, of Clifton Forge.

Wade Process Corp., New York, N. Y., has been incorporated with a capital of \$300,000 to deal in oils and greases. The incorporators are L. Vinton, C. Mason, J. M. Keating, 30 Church Street.

The West Texas Sulphur Company, a Delaware corporation, has been granted a permit to do business in Texas. The company has \$500,000 capital stock and has headquarters in Philadelphia. Texas headquarters are in Pecos.

Williams & Ferguson, Inc., Terre Haute, Ind., has been incorporated with a capital of \$15,000 to manufacture and sell wall paper, glass and oils and other articles. The incorporators are O. R. Ferguson, John Williams, Chas. F. Williams.

Worth Steel Co., Wilmington, Del., has been incorporated with a capital of \$2,500,000 to manufacture and deal in iron, steel, manganese, etc. The incorporators are J. S. Worth, W. P. Worth, E. H. Worth, W. A. Worth, N. T. Entekin, all of Coatesville, Pa.

## Construction and Operation

### Arizona

CASA GRANDE.—Plans are being prepared for erecting concentrating plant at Lake Shore mine south of Casa Grande. E. P. Ryan, Mgr.

### Arkansas

FLIPPIN.—The Marion County Zinc Company began operation on its new concentrating plant, having a capacity of 200 tons a day.

### California

BERKELEY.—The Macauley Foundry Company will enlarge its plant on land recently purchased. The Senn Concentrator Company will use a new half block recently purchased for storage purposes until work on the new factory is begun.

BERKELEY.—The Paraffine Paint Company will erect a million dollar linoleum factory, the first of its kind in the West. The factory will be devoted to the manufacture of linoleum. The cost will be \$250,000.

LOS ANGELES.—The American Trona Company has plans calling for the immedi-

ate expenditure of \$300,000. The large factory will be completed. A decision was recently handed down by Judge H. T. Dewhirst in the Superior Court of San Bernardino County favorable to the corporation in a suit brought by Harry E. Lee and others. The decision gives clear title to 25,000 acres in San Bernardino County. The company will go ahead immediately with the completion of its huge potash refining plant at the harbor.

SACRAMENTO.—Plans for building a big mill on the Pacific Coast to convert sawdust into cellulose are being completed.

SONORA.—The Dutch-App Mines Company is planning the erection of a new milling plant at the App mine. In conjunction with the mill in operation at the Dutch mine, the plant will be among the largest in California. It is probable the flotation process will be employed to a large extent, as the unit embodying this method recently installed at the Dutch mine has proved satisfactory.

STEELTON.—The Warman Steel Castings Company of Redondo Beach will build a foundry here. The company has operated an electric furnace at Redondo since 1913.

### Connecticut

BRIDGEPORT.—The Hamilton & De Lora Company will have its new factory ready by June 1, 1917, for the manufacture of metal stamps and presses. The company was recently formed, with \$100,000 capital, and has the following officers: H. H. Hamilton, president; W. E. Cutler, superintendent, and R. L. Leach, chief chemist and metallurgist.

### Illinois

CHICAGO.—The Reder Foundry Company has purchased land at the southeast corner of Oakley Avenue and 35th Place and will erect a foundry, to cost \$30,000.

CHICAGO.—The Iroquois Iron Company will erect a new blast furnace plant, which will cost more than one million dollars. Work will be started at once and will be pushed to completion by the end of the year. The plant will have a capacity of about 550 tons a day and it will increase the company's output to 1300 tons daily. The plant will be located at South Chicago, at the mouth of the Calumet River.

VANDALIA.—The Ford Manufacturing Co., manufacturers of roofing materials and compounds, will build a new lathe plant, paper mill and power plant.

### Iowa

GILMORE CITY.—Work on the 1000-barrel plant of the Fort Dodge Portland Cement Company is approaching completion, and if no further serious delays are met in the receiving of supplies this plant will be manufacturing cement by the middle of the year. Mr. H. H. Leh, formerly chief chemist for the Lehigh P. C. Company at Mason City, is to be superintendent of this new mill.

MASON CITY.—Construction on the new 1,500,000-dollar beet sugar plant of the Northern Sugar Corporation is being pushed as rapidly as possible, though some delay is experienced in getting the structural steel.

### Kentucky

JEFFERSONVILLE.—The Armour Fertilizer Company expects to start work on erecting a plant in the near future.

MIDDLEBORO.—A co-operative paper mill was proposed at a meeting of representative publishers from southeastern Kentucky and eastern Tennessee on Jan. 20.

### Maryland

BALTIMORE.—The American Refractories Company, Joliet, Ill., will erect a plant here, to cost \$100,000.

BALTIMORE.—The Canton Company is planning the erection of a \$75,000 factory at Highlandtown for the Electrotyping Zinc Company.

BALTIMORE.—Electrolytic Zinc Company, 16th St. and 2d Ave., with plant at Highlandtown, plans erecting \$75,000 plant in the same section.

BALTIMORE.—The Davison Chemical Company is contemplating erecting a large Machine shop that will cost \$100,000.

### Massachusetts

NEW BEDFORD.—The Taunton-New Bedford Copper Company will rebuild plant recently destroyed by fire, with loss of \$275,000.

### Michigan

MANISTEE.—Work on the new plant of the Filer Fibre Company, a \$300,000 concern, is advancing splendidly and is expected to be completed in three months.

REED CITY.—The Ex-Cel-O-Paint Company, capitalized at \$100,000, will begin active manufacturing operations in a short time at Marion. A new plant will be built.

### Missouri

POPLAR BLUFF.—The Butler Iron Company, recently incorporated, will erect blast furnace, concentrating plant and chemical plant to cost about \$650,000. W. W. Mechling, pres., Rookery Bldg., Chicago, Ill.

ST. LOUIS.—The Missouri Plate Glass Company, with a capital of one and one-half millions, with the Valley Park Realty Company, capitalized at half a million, have recently been incorporated and will undertake the manufacture of glass in Valley Park.

### Montana

BUTTE.—A number of capitalists have prepared plans to establish a paper pulp mill near Big Fork, in Montana. The paper pulp will be made from the Montana spruce, which abounds in the Flathead country. The incorporators of the company are E. S. Warner of the McGill-Warner Company of St. Paul; G. M. and L. S. Gillette of the Minneapolis Steel and Foundry Company; J. F. Jordan of the Wyman-Partridge Company, Minneapolis, and W. P. Snow, of Big Fork, Mont.

### Nebraska

ALLIANCE.—The Black Hills Mica Company has planned the erection of a mica refining plant here. The plant will be for splitting and grinding mica as obtained from the mines.

### New Jersey

BAYONNE.—The Texas Company has taken over about seven acres of land in Bayonne, fronting on Newark Bay, for a refinery.

LINDEN.—The American Cyanamid Company, 200 Fifth Ave., New York, has taken over the business and property of the Ammo-Phos. Company and will complete its plant at Linden. Several buildings are ready for use.

NEWARK.—The Central Dyestuff & Chemical Company, Plum Point Lane, will erect 4-story warehouse, 75 x 129 ft.; cost \$80,000.

NEWARK.—The Western Manufacturing & Oil Company, a West Virginia corporation, have sold a vacant plot to the Upson-Walton Company of New York, who will begin the erection of separate buildings for rope plant, galvanizing and warehouse purposes, covering about 30,000 square feet.

WHARTON.—The Wharton Steel Company has been bought by J. L. Repligle and will soon be placed under the direction of one of the best-known steel men. The property lies within thirty-five miles of seaport. Several millions will be spent on a new plant.

### New York

COHOES.—The Cohoes Rolling Mills have been purchased by Albany interests. Plans are under way to enlarge the output and make additions, so that ultimately 3000 men will be employed. The company is capitalized at \$500,000.

DUNKIRK.—C. A. Friederich, formerly general manager of the Dunkirk Glass plant, is making plans for the establishment of a new glass plant in this city, which will make the third glass plant to be established here since last year.

ELMIRA.—The Elmira Foundry Company, Incorporated, has secured options on a big plot of land. This property is not considered desirable for manufacturing until permission has been secured to close certain streets.

NIAGARA FALLS.—The new plant of Isco Chemical Company is operating successfully. The plant was begun on March 25, 1916, by the John W. Cowper Company of Buffalo. The cost of the plant was \$400,000, and it is located at Union St. and Royal Ave. There are nine buildings, equipped with the most modern and complete machinery and apparatus for the manufacture of caustic soda and bleaching powder. The company is connected with the Innis-Spelden Company of New York. The output is sold by this company, the principal demand outside of that in this country now being in Japan, Russia and South America. Mr. Eben C. Spelden is general manager of the company. The plant has been operating since November 10 and now operates 24 hours per day.

### Ohio

CLEVELAND.—Mr. Charles E. Sheehan of Worcester, Mass., will superintend the construction of the new \$400,000 main building of the National Malleable Iron Company, to be erected at Woodhill Road and Quincy Ave., S. E.

CLEVELAND.—The National Malleable Castings Co., 7706 Platt Ave., will erect a plant at Belt Line RR. and Woodhill RR., costing about \$250,000.

**EAST LIVERPOOL.**—The National Drawn Steel Company has made a request for a portion of the city farm for an addition to their plant. The capital has been increased from \$100,000 to \$150,000.

**MASSILLON.**—The National Pressed Steel Company began the erection of the main building on Jan. 9. The company was recently incorporated for \$1,000,000. Mr. H. M. Naugle is general manager. All the machinery and equipment has been ordered.

**NILES.**—The Basic Steel Company plans the erection of another plant in the Mahoning Valley. W. A. Taylor of Niles is president of the company.

### Oklahoma

**DILWORTH.**—Because of the shortage of natural gas in West Virginia, glass plants may go to other cities. Dilworth has 500,000,000 cubic feet of gas daily available.

**OKLAHOMA CITY.**—The Burdett Oxygen Company will begin operation of its Oklahoma plant, located at the Stock Yards Station, Oklahoma City, on Feb. 15, and will be in a position to furnish oxygen to users in that territory. This is the twelfth plant installed by the Burdett Company in the various industrial centers of the country.

### Pennsylvania

**ESSINGTON.**—Announcement has just been made by the Westinghouse Electric & Mfg. Company that the plot of ground recently purchased at Essington, near Philadelphia, will form a new industrial center for the Westinghouse Electric interests. The site embraces about 500 acres, with a frontage of approximately one mile on the Delaware River. Additional transportation facilities will be afforded by tracks from the Pennsylvania and Philadelphia and Reading Railroads.

This new center will be devoted to the production of large apparatus, the first group of buildings being for power machinery, principally steam turbines, condensers and reduction gears. The initial development will cost in the neighborhood of \$5,000,000 or \$6,000,000, occupying about one-fifth of the area of the entire plot. The group will consist of the following buildings: two large machine shops, an erecting shop for heavy machinery, forge shop, pattern and pattern-storage shop, and power house. Work will begin on these as soon as satisfactory building contracts can be let.

The number of employees to be engaged at the new plant has not as yet been definitely determined, but will number several thousand people, and undoubtedly will in the future equal the number employed at East Pittsburgh, representing over 20,000 people.

**MARCUS HOOK.**—The General Chemical Company has purchased two tracts of land, together comprising twenty-one acres, and it is understood that in addition to a plant a number of houses for employees will be built.

**NORTH PHILADELPHIA.**—The Scientific Equipment Co., 2951 N. 27th St., Phila., plans the erection of a 3-story warehouse, to cost \$200,000.

**PITTSBURGH.**—The Raymond Bros. Impact Pulverizer Co., Chicago, Ill., has sold seventeen of its largest roller mills to the Carnegie Steel Company. Representatives of the company have been in Pittsburgh during the last month installing the mills. They are used for pulverizing coal for open-hearth furnaces. Five of the mills were installed at Clairton and twelve at Homestead. Those at Clairton have been set up and put in operation, and those at Homestead are partly in operation.

**POTTSVILLE.**—The Eastern Steel Company will enlarge its plant in the spring. The company has recently completed several purchases of land.

**SHARON.**—The Knox Pressed and Welded Steel Company has enlarged its capacity by 100 per cent. The plant has only been operated a short time in its present location, but has been rushed to capacity ever since being completed.

**SHREVEPORT.**—The Caddo Oil and Refining Company have filed a mortgage here to-day in favor of the Commercial Trust Company of Philadelphia for \$10,000,000, to cover the purchase of nine local oil and refining companies. The new company will begin at once to expend \$500,000 for improvements and development of properties, which include large acreages in the Caddo and Red River districts.

### South Carolina

**COLUMBIA.**—Mr. Lloyd H. Grandy will erect a paper pulp mill plant to handle 10 cords of wood a day.

**COLUMBIA.**—The Union Bleaching Company proposes to double the present size of its plant. Improvements will represent an outlay of \$250,000.

### Tennessee

**CLEVELAND.**—The Dixie Foundry Company is enlarging its plant in order to triple its output. The company will manufacture light and medium gray iron castings.

**KINGSPORT.**—The new glass manufacturing company, which will employ about 300 men, will erect its plant this year, at a cost of \$150,000. Another company, which will manufacture oak veneer, has located here.

### Texas

**HOUSTON.**—The Sinclair Oil and Gas Corporation will build a 500-mile pipeline from the Healdton oil field in southern Oklahoma to Houston, Tex. The company will then have pipelines running from the Gulf of Mexico to the Great Lakes, the system being about 1500 miles long. A big refinery will also be built near Houston. The company has also gained the concession of 9,000,000,000 acres in Costa Rica.

### Utah

**SALT LAKE CITY.**—The Cache Sugar Company has issued \$150,000 additional stock and planned the establishment of a sugar plant at Cornish, Cache County, situated on the Utah-Idaho state line. The plant will cost about \$600,000. The company has a capital of \$800,000. Mr. J. A. Hendrickson of Logan is president; J. Will Knight, of Provo, is vice-president; Lon J. Haddock, Salt Lake, secretary.

### Washington

**TACOMA.**—The Tacoma City Council has entered into contract with Morton Gregory, of 2113 North Anderson St., Tacoma, for the lease of 3800 sq. ft. of floor space on the Municipal Dock property for one year. This space will be used by Mr. Gregory for the manufacture of synthetic rubber. He has spent considerable time in experimenting upon his formula and this last year has tested it out in various laboratories, among others at the University of Washington, where, it is claimed, one of the professors made synthetic rubber by following out his process. Work will be commenced as soon as machinery arrives from the East. The process is protected under patent rights, both in this country and abroad.

**TACOMA.**—The Bilwore Alloys Company, producers of electric furnace ferromanganese, has engaged W. K. Booth as consulting engineer. The company is enlarging its present plant at Tacoma and will manufacture ferrochrome, ferrotungsten and silico-manganese, in addition to ferromanganese. A one-ton tungsten furnace, a six-ton ferromanganese furnace and a five-ton ferrochrome furnace will be added.

**SEATTLE.**—The Rothert Process Steel Company, Joshua Green Bldg., Seattle, has contracted for the erection of four Wile electric furnaces for making steel direct from magnetite ore.

### West Virginia

**CABIN CREEK.**—The Ohio Cities Gas Company has planned the erection of a half-million-dollar plant at the mouth of Cabin Creek, near the company's big oil pool in West Virginia. The plant will be used for the manufacture of salt. The territory is in the midst of what are said to be very large salt deposits, with permanently favorable fuel supply. The plant will handle about 12,000 barrels of salt water daily and it is expected to produce 36,000 tons of salt, 130,000 pounds of bromine, 7000 tons of calcium magnesium chloride annually.

### Canada

**BRIDGEPORT.**—The Canadian Chicago Bridge & Iron Works, with headquarters at 37 W. Van Buren St., Chicago, Ill., plans the erection of another plant in the spring.

**LADYSMITH.** B. C.—The Ladysmith Smelter Company, formerly operated by the Tyee Copper Company, an English concern, has been purchased by American capitalists, and \$100,000 will be spent on improvements.

## Manufacturers' Notes

**THE BRAINARD-FAIRCHILD ENGINEERING CO.**, 330 Old Colony Building, Chicago, has been organized for the purpose of giving engineering assistance in connection with mining and metallurgical operations where crushing and grinding work is necessary. Mr. Brainard is head of the Brainard Pulverizer Co., of the same address.

**THE SEARCHLIGHT CO.** of Chicago, Ill., has consolidated with the Air Reduction Co. of New York and the Chicago

offices of the Searchlight Co. will be moved East.

**SPAIN WANTS CHEMICALS.**—The Merchants' Association of New York, through its Foreign Trade Bureau, recently made inquiry regarding the market for oils and chemicals in Spain. Reply has been received from the Consul General of the United States.

"In acknowledging the receipt of your letter of Oct. 10, with reference to extending the foreign trade of a New York firm dealing in oils and chemicals," says the Consul General, "I have to inform you that there is at present a great demand for such goods in Spain."

### Aniline Oils and Chemicals

"Aniline oils and chemicals for use in textile industries were formerly imported in large quantities and their lack is now keenly felt by many Spanish manufacturers. To give you a more concise idea of the situation I have tabulated for you a few of the important items in which this decrease is noticeable, showing the amounts imported during the first eight months of 1914 and the period just previous to the outbreak of the war, and during the corresponding period of 1916:

	1914 Metric tons	1916 Metric tons
Coconut, palm oil and vegetable table oils, except olive oil	1,045	781
Vegetable products employed in medicine not given separately	199	90
Products of the animal kingdom employed in medicine	96	39
Vegetable dyeing extracts	2,564	2,043
Mineral colors powdered or in lumps and those prepared with water	1,928	755
Colors derived from coal	839	137
Hydrochloric and sulphuric acids	209	20
Nitric acid	19	8
Sulpholeic and other similar acids	47	5
Chloride of lime and chloride of calcium	2,543	445

### Demand Is Strong

"The demand for drugs and chemicals in this district has been particularly marked during the last two months, many importers, manufacturers and dealers having applied at this office for the names and addresses of exporters in the United States. It has been said that if sufficient supplies of this class of merchandise cannot soon be received in Barcelona, several important industries will be obliged to restrict or even suspend work."

**TANTIRON NOT DUTIABLE.**—The United States Court of Customs Appeals in a test case brought by the Bethlehem Foundry & Machine Co. has found in favor of the importers in regard to a duty on tantiron.

The Collector classified the commodity as ferrosilicon, this calling for duty at the rate of 15 per cent. The Board of General Appraisers found in favor of the importers. The Government then appealed to the court for a review of the board's finding. It was shown at the trial that, whereas ordinary pig iron contained around 4 per cent of silicon, the tantiron in controversy had as much as 14 per cent. This fact, the Collector explained, warranted the inclusion of the merchandise in the ferro-silicon class.

Judge Martin, in his decision for the court affirming the board, and thereby sustaining the importers, said the evidence showed that the metal was not ferrosilicon, but essentially differed from it in character and use. The court remarked that pig iron was met with in the trade occasionally with a percentage of silicon as high as 14 per cent. It was accordingly held that, while the merchandise in controversy differed to some extent from the average pig iron of commerce in respect to the proportion of silicon it contained, it was, despite this fact, properly free under the pig iron paragraph.

**NEW HOME OF AMERICAN TOOL & MACHINE CO.**—The American Tool & Machine Co. dates the foundation of its business in 1843 and with continual enlargement has occupied various localities within the city limits of Boston. Incorporated in 1864, the shops were established at Hyde Park in 1874, and the offices for the last twenty years have been at 109 Beach Street. The company perfected and introduced the Weston centrifuge in 1865. During this twenty years the business has greatly increased and the desirability of more modern offices has led to the removal to the Rice Building at 10 High Street.



To accommodate the trade requiring immediate delivery of many productions in power transmission and machine shop requirements a warehouse has been established at 126 Purchase Street, which will be the shipping address. The new offices will be opened Feb. 1.

**EMBARGO ON CHEMICALS IN THE UNITED KINGDOM.**—The consul general at London has reported, under date of January 20 that the following chemicals are under prohibition of exportation: Sulphate of ammonia, prohibited to all destinations, ammonia and its salts, whether simple or compound (except ammonium nitrate, perchlorate, sulphate, and sulphocyanide), to all non-British destinations. These items replace the former heading, "Ammonia and its salts, whether simple or compound (except ammonium nitrate, perchlorate, and sulphocyanide)," which products were under prohibition to all non-British destinations.

**AMERICA'S GREATEST YEAR IN FOREIGN TRADE.**—American exports for 1916 reached the unprecedented total of \$5,481,000,000. According to a statement issued to-day by the Bureau of Foreign and Domestic Commerce, of the Department of Commerce, this exceeds the total for 1915 by \$1,926,000,000 and the total for 1913 by \$2,597,000,000. The exports for December are announced as \$521,000,000, which exceeds the previous high monthly total by \$5,000,000. The December average for the five years previous was \$263,000,000.

Imports in 1916 aggregated \$2,392,000,000, also a record total. For 1915 the total was \$1,779,000,000, and for 1912, the previous record year, \$1,818,000,000. December imports were valued at \$205,000,000, indicating a continuation of the recovery which set in during September last following the sharp decline from the large total of \$246,000,000 for June. The December, 1915, total was \$172,000,000 and the December average from 1911 to 1915 inclusive, \$153,000,000.

The year's export balance was \$3,089,000,000, as compared with \$1,776,000,000 for 1915 and \$2,456,000,000 for the five-year period from 1910 to 1914 inclusive. The December favorable trade balance was \$316,000,000, compared with \$187,000,000 for December, 1915, and \$131,000,000 for December 1914.

**NITRATE OF SODA SHIPMENTS IN 1916.**—The total shipments of nitrate of soda to American ports for 1916 was 1,225,637 tons, as against 772,190 tons for 1915.

**SCHAEFFER & BUDENBERG ESTABLISHES A ST. LOUIS OFFICE.**—The Schaeffer & Budenberg Mfg. Co. of Brooklyn, N. Y., makers of Columbia recording instruments, announce the establishment of a selling office in St. Louis, Mo., under the direction of A. H. Reuter, who was formerly connected with the Chicago office. The principal selling offices of this company are now Brooklyn, N. Y.; Philadelphia, Pa.; Pittsburgh, Pa.; Chicago, Ill.; St. Louis, Mo.; Los Angeles, Cal.

**TRAYLOR ENGINEERING CO. OPENS CHICAGO OFFICE.**—The Traylor Engineering & Manufacturing Co., of Allentown, Pa., has opened a Chicago office at 1414 Fisher Building. The office will be in charge of L. J. Hewes, who for the past thirty-six years has been intimately identified with the stone crushing machinery business of the mid-West, and for the past twelve years has been representative of the Power & Mining Machinery Co. in that territory.

**MACKINNON, HOLMES & CO. CHANGES MANAGEMENT.**—Changes in the management of MacKinnon, Holmes & Co., Ltd., of Cherbrooke, Que., have recently taken place, caused by the retirement from the company of A. R. Holmes, who in the past has occupied the position of director and secretary-treasurer.

It is understood that J. W. Bowman, president, and G. D. MacKinnon, vice-president and general manager, have purchased the holdings of A. R. Holmes and his friends, and new directors in the persons of Dr. A. W. Klein of Greenwich, Conn.; M. L. MacKinnon and J. Nicol of Sherbrooke, Que., have been elected, with F. C. Johnston, secretary-treasurer.

The business will be continued as in the past under the management of G. D. MacKinnon, and it is understood the company is making extensive plans for future development.

This company has been particularly successful in its general business of structural steel and steel plate work, having one of the most complete plants in the country for these special lines. It has also been successful in the forging of shells for the Imperial Munitions Board, having a very complete and up-to-date plant for this special work.

**MERRIMAC TAKES OVER COCHRANE CO.**—The formal transfer of the plant and business of the Cochrane Chemical Company to the Merrimac Chemical Company has taken place, the latter concern taking over also the personnel of both office and factory management of the Cochrane company, so that the business of the Cochrane company will continue without interruption. Business, however, will be carried on in the name of the Merrimac Chemical Company and all communications should be made to it at 40 Central Street, Boston, Mass.

**SEVERAL DIRECTORS RESIGN FROM FEDERAL DYESTUFF CORPORATION.**—George T. Bishop, president of the Federal Dyestuffs & Chemical Corporation, resigned this week from that office. George H. Schuler, assistant to Mr. Bishop, resigned at the same time in company with four directors. The resigning directors were Ralph L. Fuller, E. G. Tillotson, Mark W. Potter and George A. Coulton. It is stated that other interests require the attention of these men. Mr. Bishop is now connected with Ralph L. Fuller & Co., of which Mr. Fuller is the president.

**BIG YEAR IN OIL INDUSTRY.**—At the annual meeting of the Standard Oil Co. of New Jersey, F. W. Weller, vice-president, said: "Owing to unprecedented demand for petroleum products 1916 has been a prosperous year in all branches of the business. During the year total production exceeded that of 1915, but the growth in demand was greater than increased production, and this situation is being reflected by advancing prices in crude. The brisk demand for the past two years has so stimulated construction of refining facilities that this capacity is now in excess of production of crude. Outside capital has been used largely in providing this excess and when the lean years come, as they do with great regularity in this business, this new capital will be brought to a realizing sense that it is uneconomic to pay fancy prices for either production or refining capacity. However, concerns that have been conservatively operated will probably on account of the demand continue to enjoy prosperity during the current year."

**THE FIRST NATIONAL BANK OF BOSTON OPENS BUENOS AIRES BRANCH.**—Arrangements have been completed for the opening by this bank of a branch in Buenos Aires under the management of Noel F. Tribe, a banker of experience, who has resided in the Argentine for the past twenty years and is exceptionally well versed in South American financial and trade conditions. Mr. Tribe will return to Buenos Aires at the end of February and during the coming month will be very glad to meet at the bank or correspond with those who may care to take advantage of the opportunity to discuss with him details of South American business.

This branch was established to assist in building up the foreign trade between the United States and Argentina. As so much of this trade passes through the port of Boston, and as New England exporters and importers are so vitally interested in accurate foreign credit information and dependable financial arrangements, it seems appropriate that this bank extend its activities in this way for the general benefit of our foreign trade.

The First National Bank of Boston is already prominent in financing the foreign trade in the foreign field, having direct bank correspondents in all the financial centers of the commercial world. Due to these connections, which its foreign department has built up in the last fifteen years, soon after the war broke out it took the lead in the volume of foreign business transacted, leading all other banks in this respect.

The advantage of a direct branch in the Argentine is obvious not only to assist in building up foreign trade during the present unsettled trade conditions, but also as a matter of preparedness to assist American exporters to retain as large a percentage of present business as possible after the close of the war.

**THE GULICK-HENDERSON COMPANY,** consulting and inspecting engineers, announce the removal and consolidation of their general offices from 30 Church Street and 120 Broadway, New York, to 13-21 Park Row.

**NITRATE SHORTAGE FEARED BY GREAT BRITAIN.**—England fears a shortage of nitrate of soda unless large shipments are soon forthcoming from Chile. W. D. Montgomery & Co., of London, have published a review of the situation for the last half of 1916.

After noting the upward course of prices in Chile during the past six months, they say:

"The fact that even 1918 has shared in the business done at 8s 3d ordinary, and 8s 6d for refined per quintal, is strong evidence that, in some quarters at least, a hopeful view is taken of the industry when, presumably, war will be no more. We have no valid reason for condemning such a policy, but it might perhaps be just as well to exercise caution in the matter of far forward purchases, for who knows what new features in the industry the necessities of war may have brought about. It is tolerably certain that, even if no new discovery has been made, there will be a great deal of machinery for the manufacture of synthetic nitrate in Germany which may become, on the basis of its new capitalization, a formidable competitor, for a time, to the Chilean article. Perhaps, however, the prices now obtaining for 1917 are almost more dangerous from a purchaser's point of view (apart from Government purchases), being relatively so much higher. German peace offers must tell upon the nerves of holders and producers when prices rise to giddy heights. So far as the first six months of the year are concerned, we think holders have little to fear unless they have failed to secure their freight, but afterwards there might be more danger, having in view the latest move on the part of Germany to bring about negotiations for peace.

"Twelve months ago some doubt existed as to the capacity of the market to cope with the increase which was taking place, but this fear has been set at rest during this past few months by the market's wonderful power of absorption. The question is, how long will it last? Should the present rate of production be the maximum, it is hardly likely that it will be found to be too much until the war is over, and that is about all one can say.

"The figures of production for the year are 63,300,000 quintals, against 38,123,000 the previous twelve months. Stocks in Chile are to-day just about what they were a year ago, being about 16,300,000 quintals, against 17,099,000. Freight to-day are very nominal, quotations being about 140s to 150s per ton for sail or steam, but it is doubtful if one would be forthcoming at even 160s.

"The conclusions to be arrived at from these considerations is that nitrate in the immediate future cannot well be cheaper than it is to-day and it may very easily be dearer, nevertheless, barring sulphate of ammonia, which may only be obtained in small doses, if indeed it can be had at all, it is probably cheaper than any other form of fertilizer obtainable. The pity is that supplies are so slender."

## Manufacturers' Catalogs

**DENVER ENGINEERING WORKS COMPANY,** Denver, Colo., have issued Bulletin No. 1077, January, 1917, dealing with the Dewco ball mills.

**MANITOWOC ENGINEERING WORKS,** Manitowoc, Wis., has issued a pamphlet describing the Manitowoc dryer.

**THE SIDCO COMPANY OF AMERICA, Inc.,** 37-39 Murray Street, New York City, have issued a little booklet "Pure Fused Silica," describing their products, which are Made in America.

**THE NATIONAL CHEMICAL COMPANY,** People's Gas Building, Chicago, Ill., have issued a booklet on potash manufacture from kelp, dealing in its uses, industry and commercial value.

**MESTA MACHINE COMPANY,** Pittsburgh, Pa., have issued Bulletin "D," a booklet on the Mesta automatic plate valves (Iversen Patent).

## Other New Publications

**THE RADIUM-URANIUM RATIO IN CARNOTITES.** By S. C. Lind and C. F. Whittemore. Technical Paper 88, Mineral Technology 6, issued by the Department of the Interior, Bureau of Mines.

**OUR ANALYTICAL CHEMISTRY AND ITS FUTURE.** Chandler lecture at Columbia University. By William Francis Hillebrand, chief chemist of the Bureau of Standards, at Washington, published by the Columbia University Press, New York. Agents, Lemcke & Buechner, New York.

**BIBLIOGRAPHY OF RECENT LITERATURE ON FLOTATION OF ORES.** Compiled by D. A. Lyon, O. C. Ralston, F. B. Laney and R. S. Lewis. Technical Paper 135, published by the Department of the Interior, Bureau of Mines, January to June, 1916.

**UNITED STATES GOVERNMENT SPECIFICATION FOR PORTLAND CEMENT.** Third edition issued Jan. 18, 1917. Circular No. 33 of Bureau of Standards.